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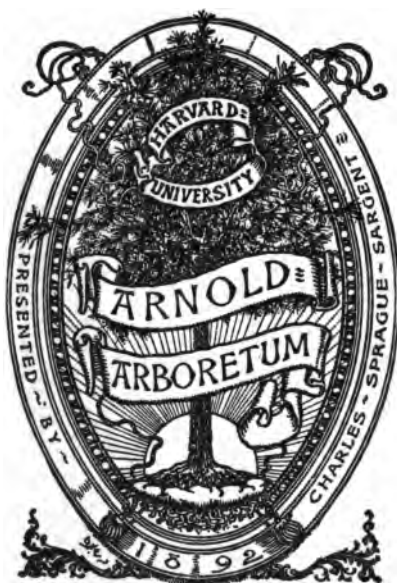
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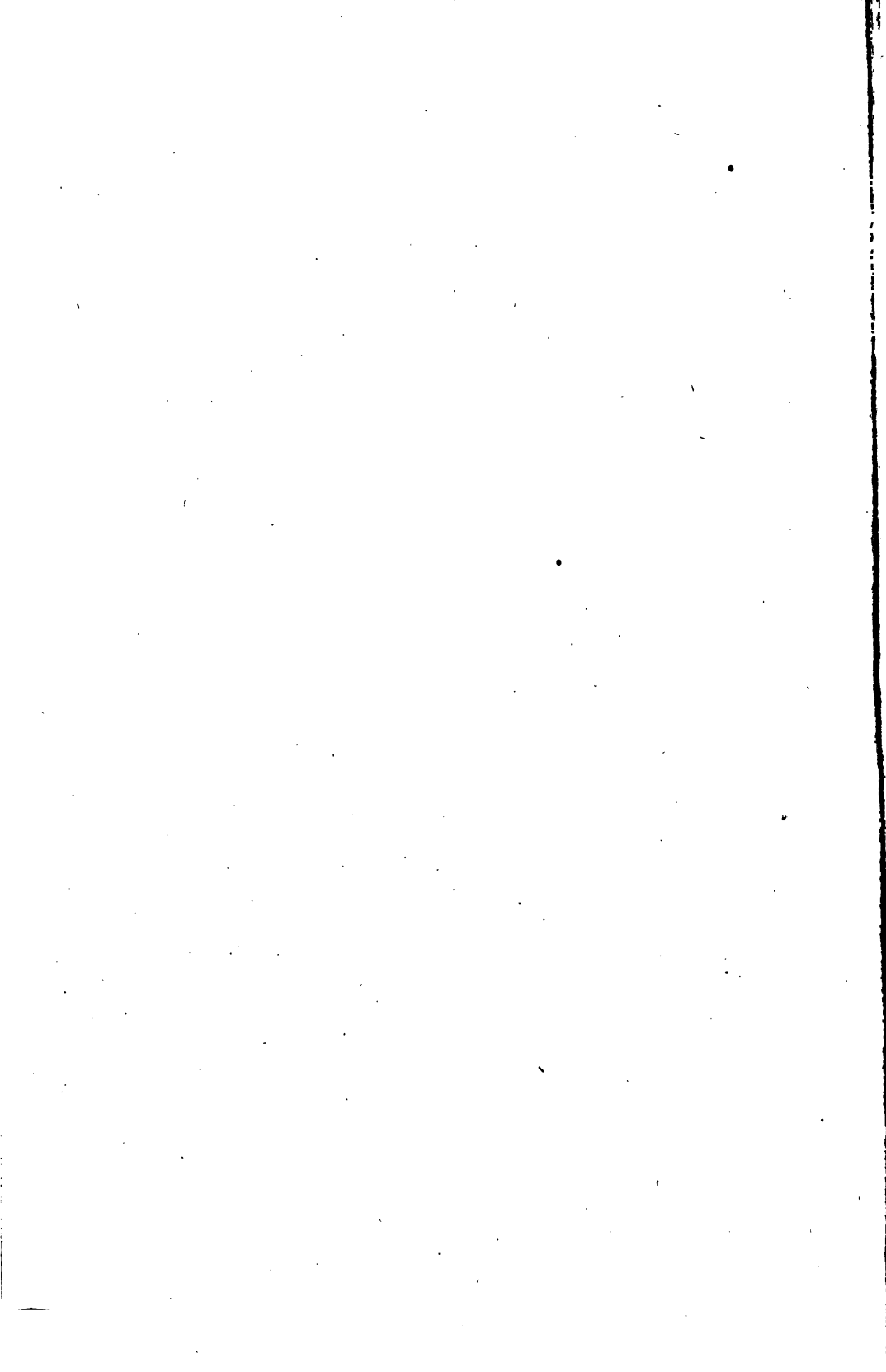
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BUREAU OF CHEMISTRY—BULLETIN No. 135.

H. W. WILEY, Chief of Bureau.

COMMERCIAL TURPENTINES:

THEIR QUALITY AND METHODS
FOR THEIR EXAMINATION.

BY

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Chief, Leather and Paper Laboratory,

AND

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1911.

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LETTER OF TRANSMITTAL

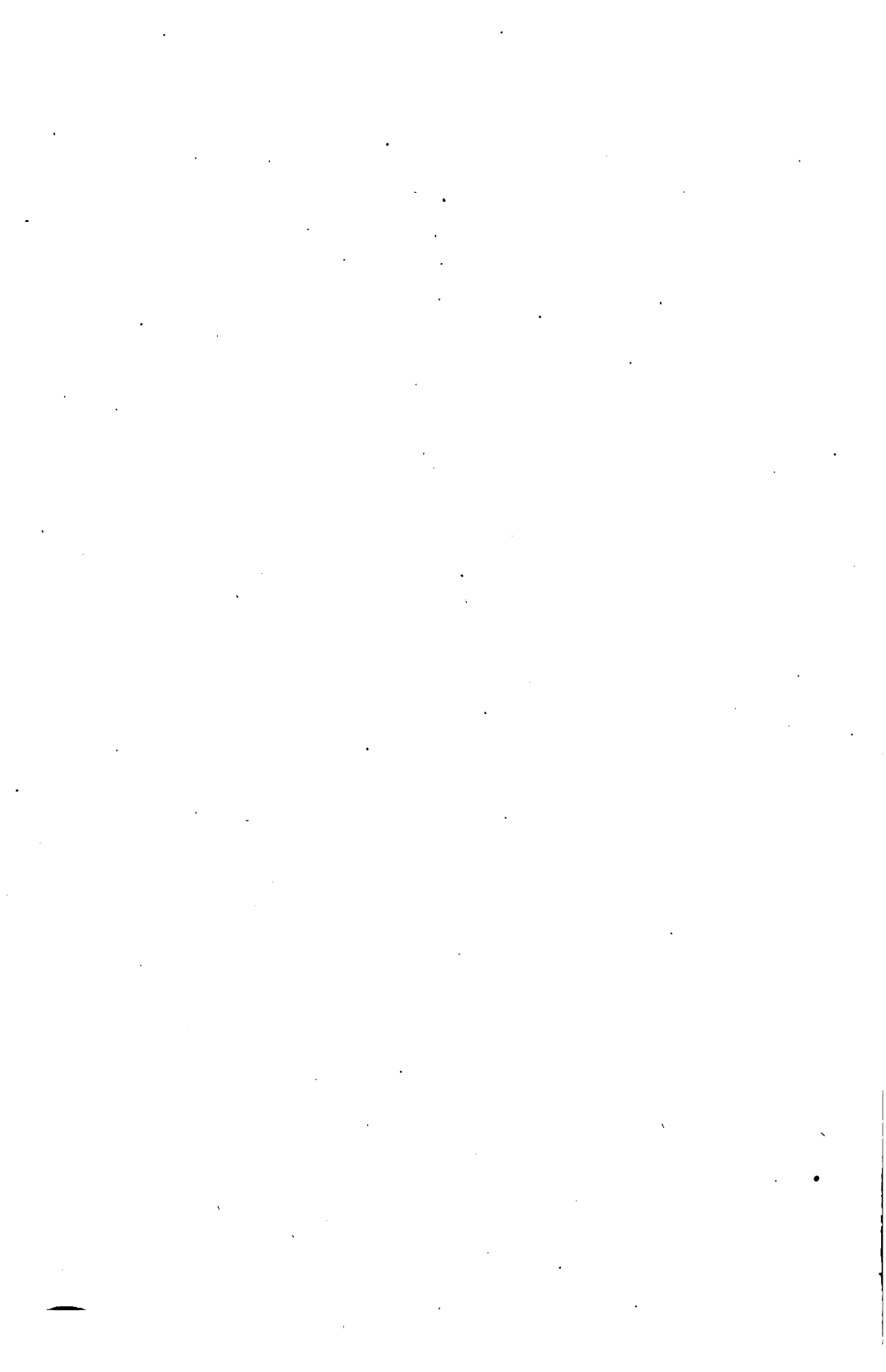
U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF CHEMISTRY,
Washington, D. C., October 15, 1910.

SIR: I beg to submit for your inspection and approval the results obtained in an investigation made in this Bureau by F. P. Veitch and M. G. Donk on the adulteration and grading of turpentines. The results show the extent to which turpentine is adulterated, and indicate the loss of values to the turpentine farmers, and the extent to which turpentine users are defrauded by adulteration. Methods for the analysis and testing of turpentine have been simplified and improved. Based largely on this investigation and the field work connected therewith, specifications for grades are suggested which it is believed will prove equitable to the producer and enable the purchaser to secure turpentine of the character desired. The data obtained are thus of special interest to the buyers and sellers of this product. I recommend that this report be published as Bulletin No. 135 of the Bureau of Chemistry

Respectfully,

H. W. WILEY,
Chief of Bureau.

Hon. JAMES WILSON,
Secretary of Agriculture.



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COMMERCIAL TURPENTINES.

INTRODUCTION.

NATURE AND GENERAL PROPERTIES.

Commercial spirits of turpentine is a mixture of similar and closely related terpenes having the general composition expressed by the formula $C_{10}H_{16}$, with varying quantities of oxidized and hydrated derivatives of terpenes, usually present only in small amounts when first distilled from the gum.

These terpenes differ both in their physical and chemical properties, and consequently commercial turpentine may distill at from 154° to 215° C. (309° to 419° F.). The fractions obtained on distillation have specific gravities ranging from 0.8580 to 0.9500 or more, with refractive indices of from 1.462 to 1.520, respectively.

The pinenes, several of which, differing slightly from one another in boiling point, specific gravity, and refractive index, have been recognized, are among the chief constituents of spirits of turpentine freshly distilled from the gum. Dipentene¹ has also been reported as occurring in American turpentine, but as dipentene distills alone at about 178° C., it is probably present in but small quantities in those turpentines which distill completely below this temperature. The above-mentioned terpenes boil between 155° C. (311° F.) and 178° C. (352° F.); their specific gravities lie between 0.845 and 0.866, and their refractive indices between 1.46 and 1.472. Other constituents which may be present, regarding the identity of which but little is known, have specific gravities of from 0.865 to 0.950 and refractive indices as high as 1.5200 at 20° C., and boil at from 160° C. to 215° C. (320° to 419° F.). Turpentines which have been kept long, particularly those held in partly filled vessels, frequently have higher specific gravities, refractive indices, and boiling points than freshly distilled turpentine. In changing they become more oily and usually somewhat darker in color.

The quantity of each constituent present in commercial American turpentine is influenced not only by the physiological processes of the individual trees, but also, and probably more largely, by the

¹ Gildemeister, *Die Aetherische Oele*, 2d ed., 1910, p. 328; Allen, *Commercial Organic Analysis*, 3d ed., vol. 2, pt. 3, p. 263.

conditions under which the crude gum is gathered and the time which elapses before it is distilled, by the method of conducting the distillation, and finally by subsequent storage conditions. When the face of the tree over which the crude gum flows before it reaches the box is long, the weather hot, and the gum is kept long before it is distilled, there is more evaporation of the lighter constituents and at the same time the oxidation and polymerization of the gum are increased. It is customary to carry the distillation further on gum from old than from new boxes. The temperature in the final stages of the distillation rises higher, as a rule, and the product obtained near the close of the distillation is more frequently colored or burned. For these reasons turpentine from old boxes, as a rule, is heavier, distills less completely below 170°C. , and has a higher refractive index than turpentine made from first-year gum or "virgin dip."

One of the chief causes of the observed variations in the constants of turpentine freshly distilled from the gum lies in the practice followed in the barreling of the product at the still. From time to time during the distillation the turpentine is transferred to barrels for shipment, and as a rule the turpentine first distilled is placed in one barrel, the middle portion in another, and the last off in a third barrel. In the earlier stages of the distillation the turpentine having the lowest specific gravity, refractive index, and distilling temperature passes over. As distillation proceeds the distillation temperature, gravity, and refractive index of the distilling turpentine steadily increase, though with occasional lapses and not necessarily proportionally. As a result the constants of the turpentine in the first barrel may agree with the lower limits, while that in the last barrel may approach the upper limits recognized for turpentine.

Because of these conditions, exposure in the woods, variations in distilling, differences in the product at successive stages of the distillation, and changes in composition after distillation, it is customary to recognize variations in the specific gravity of turpentine from 0.862 to 0.875, in refractive index from 1.4670 to 1.4750, both at 15.5°C. , and in the initial boiling point of from 154°C. to 159°C. (309° to 318°F.), while the percentage which distills below 170°C. (338°F.) varies from about 85 to 99 per cent.

Old turpentine, even though it is kept in closed vessels, may undergo changes and have a specific gravity as high as 0.9460 and a refractive index of 1.480 or more, while the initial boiling point is raised and the percentage distilling below 170°C. (338°F.) is lowered.

It is desired in this bulletin to distinguish between old turpentine and turpentine to which mineral, coal tar, or other dissimilar oils have been added. While old, highly oxidized turpentines can not properly be classed as normal turpentines, they are, nevertheless,

mixtures of terpenes and related heavier oils and as such may possess the peculiar properties which are commercial assets of turpentine and which distinguish it sharply from unrelated oils which are often substituted wholly for it or employed in its sophistication. In the one case the product is a natural result of aging, while in the other it may properly be said to be adulterated, and such oils will be so distinguished in this publication.

SOURCES AND METHODS OF PRODUCTION.

Oil or spirits of turpentine, commonly known as "turpentine," or in the paint trade simply as "turps," is obtained chiefly from the longleaf pine (*Pinus palustris*), though a portion is also obtained from Cuban (*P. heterophylla*) and a little from loblolly pine (*P. tæda*). The Forest Service has found that loblolly, shortleaf, and Virginia pines yield naval stores equal in quantity to the longleaf yellow pines, and as the former occur in large quantities in the South their utilization would add largely to the turpentine resources of the country.

During the winter months before the sap begins to rise a pocket or "box" is cut in the tree near the ground, and at intervals during the succeeding spring and summer the bark and a portion of the sap wood are removed from the trunk above the box. This cutting greatly shortens the life of the tree and renders it more liable to destruction by wind, storm, and fire. The injury to standing timber caused by turpentering may be largely avoided by collecting in cups, the gum being directed into them by strips of zinc or heavily galvanized iron set in the face of the tree. This system, used exclusively in France, is now being extensively introduced in a modified form into this country and not only protects the tree but, as has been shown by the Forest Service, yields a higher grade of rosin and more turpentine.

In the spring the crude gum begins to flow from the "face," as that portion of the tree from which the bark and a part of the sapwood have been removed is called, and collects in the box or cup near the base, from which it is removed by dipping from time to time during the flowing season, which lasts from March to October or later. The thick, resinous mass, which usually contains water, chips, bark, pine needles, earth, and charred wood, is taken to the still, which is a large copper kettle with an outlet in the bottom by means of which it may be emptied and a cap connecting with a condensing coil. The kettle is bricked around in order that it may be safely heated by a fire underneath.

The crude gum is poured into the kettle until it is from one-third to one-half full and then slowly heated. A portion of the chips, bark, and pine needles is skimmed out after the contents of the kettle

become thoroughly liquid, and the cap is replaced in position and connected with the condenser while the heating of the kettle is slowly continued. Slow, cautious heating is made necessary by the presence of water in the gum, which causes frothing when the gum begins to boil, and if the heating is unduly hastened the gum will froth over, causing loss and delay. The presence of water in the gum lowers the boiling point of the mixture, so that both turpentine and water begin to distill at about 94° to 95° C. (201° to 203° F.); alone it distills at from 155° to 170° C. (311° to 338° F.).

When the danger of frothing over has passed, which means usually that most of the water has boiled off, a small stream of water is allowed to run into the still through the cap, as in this way the distillation is conducted at a much lower temperature than would otherwise be possible.

The condensed turpentine and water are collected in a separator, where the turpentine collects on top and may be drawn off from time to time. The separated spirits of turpentine is then run into tight white-oak barrels, which have been well driven and glued to prevent the turpentine soaking through the wood. The barrels are filled to within 1 gallon of their capacity, leaving room for expansion of the turpentine, which takes place with a rise of temperature and would cause leakage if the barrel were filled at the ordinary temperature. The barreled turpentine is then ready for the market.

The residue in the kettle from the turpentine distillation is rosin, which while still hot is run out of the opening in the bottom through wire screens and then strained through cotton cloth or batting to remove the remaining chips, straw, and dirt, after which it is barreled and cooled.

When distillation is properly conducted with water all of the turpentine distills below 150°-160° C. (302°-320° F.), and there is little apparent breaking up of rosin or discoloration of the turpentine. If the temperature is allowed to rise above 160°-170° C. (320°-338° F.) toward the end or the distillation is prolonged, or if water is not used in distillation, both the turpentine and the rosin are darker colored.

The color of turpentine is due primarily to overheating the gum and possibly partly to the chips, pine straw, etc., left in the kettle during the distillation. Wood begins to color decidedly above 150° C. (302° F.), so that it is almost impossible to make a colorless rosin if there are any chips or bark in the still. Prolonged heating of the rosin at 130° C. will lower the color several grades. Hence, to make turpentine and rosin having but little color the gum should be free of wood, bark, and pine straw, the contents of the still should never rise above 160° C. at any point, and the distillation should be conducted as rapidly as is practicable.

In the past but few stills were equipped with thermometers to indicate the temperature of the gum in the still, but it is probable that in properly conducted distillation the temperature rarely rose above 170° C., and for virgin gum was considerably lower than this. Thermometers are coming into more general use in turpentine distilling, and the danger from overheating is being correspondingly reduced.

DISTRIBUTION, QUANTITY PRODUCED, AND VALUE.

The turpentine-producing area in this country is practically confined to the coastal plains region of the Southern States. In the earlier days the industry was best developed in North Carolina, but owing to destructive methods of turpentine orcharding, in conjunction with lumbering, fires, etc., the industry has gradually worked southward and westward, until at present Florida produces the most turpentine, followed by Georgia, Alabama, Mississippi, Louisiana, North Carolina, South Carolina, and Texas, in the order named.

The statistics of the production for the past 40 years taken from the census reports are given in the following table:

Turpentine production in the United States for the past 40 years.

Year.	Gallons.	Value.
1870.....	6,004,887	\$2,194,498
1880.....	17,565,250	1,542,120
1890.....	17,316,200	5,459,115
1900.....	38,488,170	14,960,235
1905.....	30,687,051	15,170,499
1908.....	36,589,000	14,112,400

GRADING, MARKETING, AND USES.

It is the trade practice to grade turpentine according to its color, and the various grades are known as "water white," "standard," "off one shade," "off two shades," and "off three shades." The latter is not merchantable. Under the trade regulations the deduction in price on turpentine off one shade is 2.5 cents per gallon and off two shades 4 cents per gallon. Of late years, however, it has become customary to mix the colored turpentine with water-white or standard turpentine, adding a small quantity to each barrel which is not full. The factor is paid 50 cents for each barrel of the colored turpentine that he uses in this way. By this practice the producer receives more for his colored turpentine than he otherwise would under the trade regulations, and but one grade of turpentine is generally known to the buyer beyond the primary markets. There are, however, as has been indicated in the discussion on general properties and methods of production, decided differences in the

quality of various lots of turpentine. Recognizing this fact many users of large quantities buy on definite and rather strict specifications, and it would probably be of advantage to the trade if several grades for turpentine were more generally recognized.

About half of the turpentine produced in this country each year is exported, chiefly to Europe, while by far the larger part of the remainder is consumed in the varnish and paint industries, and small quantities are used in medicine and as a solvent for gums, rubber, fat, and waxes, in this country. No statistics are obtainable showing the quantities consumed for the latter purposes, nor in thinning paints and varnishes at the time they are actually used. The distribution of turpentine produced in 1905¹ was as follows:

	Gallons.
Produced.....	30,687,051
Exported.....	15,894,813
Used in manufacture of paints and varnishes.....	7,160,774
Used in medicine, and as a solvent in other industries and for paint and varnish thinning, etc. (by difference).....	7,631,464

COLLECTION AND CLASSIFICATION OF SAMPLES.

The turpentine found on the American market is quite frequently adulterated with cheaper and inferior oils, those most commonly employed being the petroleum oils having specific gravities corresponding closely to that of turpentine, and distilling at about the same temperature. Other adulterants are certain coal-tar oils, rosin spirits, and wood turpentine, which closely resemble turpentine in specific gravity, distilling temperature, and some other properties.

Work on turpentines found on the American market was undertaken under the Food and Drugs Act of June 30, 1906, for the primary purpose of determining the nature and extent of adulteration in turpentine sold for medicinal purposes. Furthermore, it is desirable to know at what stage in the marketing of this article it is generally adulterated. The naval stores industry is vitally interested in the matter, as the value of the turpentine output is materially affected by the practice of adulteration. It is quite common for druggists, both wholesale and retail, to buy turpentine from oil and paint dealers or from anyone handling it in large quantities, and, therefore, samples have been secured from oil and paint dealers, as well as from druggists, their stock being in many instances representative of the turpentine sold at the drug stores in the same community. In fact rarely is any distinction made between turpentines for drug or technical purposes.

Though it is known that spirits of turpentine is very frequently adulterated, no systematic investigation of the subject in this

¹ Later statistics on the quantity used in manufacture of paints and varnishes are not available.

country is on record. For this reason, the results obtained on samples collected from all parts of the country are recorded for the information of the public, to show the extent of the practice of adulteration, and to furnish more complete data for the grading of American turpentine and the preparation of specifications therefor which shall be fair to the producer and also insure that the consumer secures the article for which he pays.

The samples have been classified into those secured within and without the turpentine-producing States, and each of these classes has been subdivided into three groups. First, samples taken from the producer, or in the yards of primary buyers, but which the primary buyers stated, and the evidence indicated, were in the original containers in exactly the condition in which they were received from the producer. Second, samples taken from the tanks or other containers of primary buyers after they had accepted the goods from the producers. Third, samples taken from the stock of wholesale and retail druggists, paint and hardware merchants, and other dealers. These classifications are made to show as clearly as possible at what point in the turpentine industry adulteration is most practiced.

METHODS OF ANALYSIS EMPLOYED AND THEIR DISCUSSION.

GENERAL EXAMINATION.

Owing to the nature of turpentine, there are quite wide variations in its chemical and physical constants, and as these natural variations do not appear within limits to affect its suitability for technical and medicinal purposes, rigid control of analytical procedure usually is not insisted upon in the examination of the article, and, indeed, great refinement of procedure is scarcely warranted. Thus specific gravity and refractive index need not be expressed beyond the third decimal, flash point and distilling temperature should be stated in whole degrees, while the residues from polymerization and the results of distillation should be given in whole numbers as percentage. For these reasons, and in order that the results might be comparable with other published data, the usual procedure in the examination of turpentine has been followed in the main in analyzing the samples reported in this bulletin. It became apparent, however, as the work progressed that certain precautions and modifications in making such examinations were desirable in order that concordant results might be obtained by different analysts, and conditions affecting the results on adulterated and certain oxydized samples be better controlled. The details of the modified methods are given, beginning on page 26.

The methods as applied to the samples analyzed in this investigation are as follows:

Specific gravity was determined with the Westphal balance at 20° C.

Refractive index was determined at 20° C. with the Zeiss refractometer.

Color was determined by means of the Lovibond colorimeter, and is, for simplicity of standardization, expressed in terms of the depth in millimeters of the liquid which gives a color equal to the Lovibond tintometer glass No. 1 yellow.

Distillation.—One hundred and fifty cubic centimeters of turpentine were put in a 200 cc flask having a neck 20 mm in diameter and the side tube 10 cm from the bulb. The bulb of an accurate thermometer was placed opposite the side tube, the flask set in a glycerin or oil bath, connected with condenser, heated, and distillation conducted at the rate of two drops per second.

The results obtained on the commercial samples have not been corrected for the emergent stem of the thermometer, and for this reason the recorded readings are on the average, by actual comparison, about 3.5° lower, and as a maximum on the higher boiling fractions, about 6° lower, than the actual temperatures. Neither has correction been made for variation in barometric pressure. The most serious consequence of not making these corrections is found in determining the exact initial distilling point and the percentage of turpentine distilling at successive temperatures. As a matter of fact, these corrections would have but little effect in the case of a normal turpentine on the total percentage distilling below the upper limit for turpentine, which may very properly, as the work indicates, be put at 170°, when the reading is taken with the scale of the thermometer entirely within the neck of the distilling flask.

EFFECT OF EMERGENT THERMOMETER.

The differences in temperature readings, due to emergent stem, and the effect of these differences on the quantity of turpentine distilled at various temperatures as determined by actual comparisons, are shown in the following table. In all cases the bulbs of the thermometer were opposite the side neck of the flask.

Comparison of differences of temperature and of volumes distilled, due to emergent stem of thermometer.

[200 cc of turpentine distilled.]

Turpentine No. 1.				Turpentine No. 2.			
Reading of thermometer—		Difference.	Volume distilled.	Reading of thermometer—		Difference.	Volume distilled.
Immersed. ¹	Emergent.			Immersed. ¹	Emergent.		
°C.	°C.	°C.	cc.	°C.	°C.	°C.	cc.
157.0	153.5	3.5	156.0	151.0	5.0
157.4	154.0	3.4	7	158.0	154.0	4.0
158.2	155.0	3.2	26	159.2	155.0	4.2	12
160.0	156.5	3.5	108	160.2	156.5	3.7	28
164.0	160.0	4.0	166	165.0	162.0	3.0	83
165.0	162.0	3.0	173	167.0	163.0	4.0	99
170.0	166.0	4.0	183	169.4	165.0	4.5	131
172.0	167.0	5.0	184	170.0	166.0	4.0	136
.....	175.0	170.0	5.0	151
.....	177.0	172.0	5.0	155
.....	180.0	174.0	6.0	159
.....	185.0	178.0	7.0	163
.....	190.0	183.0	7.0	166
.....	195.0	187.0	8.0	168
.....	200.0	192.0	8.0	170

¹ Thermometer immersed in vapor only.

The table shows that No. 1 turpentine begins to distill at 157° C. (315° F.) by the immersed thermometer and at 153.5° C. (308° F.) by the emergent thermometer. At 160° C. (320° F.) by the immersed, 108 cc had distilled, and by the emergent thermometer 166 cc. At 165° C. (329° F.) by the immersed thermometer 173 cc had distilled, while about 180 cc had passed over when this temperature was reached by the emergent thermometer. It will be noted also that as the temperature rises, the difference between the readings of the two thermometers first decreases and then increases; the difference in reading being least where approximately the largest volume of distillate is yielded. These facts are brought out still more strikingly in the distillation of turpentine No. 2. The lag of the emergent stem thermometer is also shown in the comparison of the initial distilling temperature. The calculated correction for the emergent stem thermometer at 160° is 2°, while the actual correction as determined by experiment was never less than 3°. These figures show how necessary it is, if comparative data as to the progress of a distillation are desired to have the stem of the thermometer entirely immersed in the vapor, and they also indicate that corrections made under ordinary laboratory conditions for emergent stem and lag are likely to be inadequate.

EFFECT OF VARIATIONS IN ATMOSPHERIC PRESSURE.

Variations in atmospheric pressure may affect the volume of the distillate at any given temperature even more than errors introduced by reading temperatures on emergent thermometers. The effect of variations in pressure on the distillation of two turpentines is shown in the table on page 16.

Variations in amounts of turpentine distilled caused by different barometric pressures.

[200 cc of turpentine distilled.]

Turpentine No. 1.				Turpentine No. 2.			
Temperature.	Amounts distilled at different barometric pressures.		Difference.	Temperature.	Amounts distilled at different barometric pressures.		Difference.
	740 mm.	780 mm.			740 mm.	780 mm.	
° C.	cc.	cc.	cc.	° C.	cc.	cc.	cc.
157.4	7			159.2	12		
159.0	53			160.0	26	12	14
160.0	108	10	98	165.0	90	60	33
165.0	173	163	10	170.0	136	120	13
170.0	183	180	3	175.0	151	143	8
				180.0	159	156	6

These results show that in the early stages of the distillation the quantities distilled at the two pressures differed widely; more widely on the turpentine distilling at the lower temperatures and within the narrower limits. As distillation approached completion these differences decreased, becoming negligible when 90 per cent of No. 1 had distilled, but remaining large when 80 per cent of No. 2 had passed over.

The distillation of normal fresh turpentine, of which more than 80 per cent passes over below 165° C. (329° F.), is greatly affected by variations in pressure; and it is quite clear that in obtaining the initial distilling temperature and other distillation data at temperatures below 165° C., the pressure should always be the same. As distillation proceeds and the temperature rises the effect of variations in pressure on the total amount of turpentine distilled becomes less, so that ordinarily (that is, on a turpentine 90 per cent or more of which distills below 170° C.) when the distilling temperature rises to 170° C. the total quantity distilled at 740 mm or at 780 mm is almost the same, and for all practical purposes the difference may be disregarded. When the percentage distilling above 170° C. is large, however, as in the case of No. 2, the difference due to the variation in pressure is large and not to be ignored. If it is desired, therefore, to determine the percentage distilled below 170° C. to determine accurately the initial boiling point, or to trace the course of the distillation throughout its entire range, the barometric pressure should always be corrected to 760 mm.

DETECTION OF ADULTERANTS.

The majority of the samples analyzed were examined only for mineral oils, as these are the principal adulterants employed. Methods for the detection of certain other adulterants were studied, however, and the results obtained will be discussed. Wood turpentine was not detected in any of the samples examined.

SIGNIFICANCE OF ODOR, COLOR REACTIONS, AND BEAD.

Straight wood turpentine is readily distinguished from gum turpentine by their odor, or when they have been very carefully refined, by the odor of the first fraction, or of the residue from fractional distillation. One or both of these portions have the peculiar "sawmill smell," and the residue has a camphoric and somewhat nauseating odor characteristic of wood turpentine, which is quite different from the mild, sweet fragrance of gum turpentine.

Destructively distilled wood turpentine and also rosin spirits are more readily distinguished from gum spirits by their odor than is wood turpentine prepared by steam distillation, and they also give distinguishing color reactions when mixed with sulphurous acid as suggested by Hertzfeld or with hydrochloric acid (the presence of tin or zinc is not necessary) as suggested by Grimaldi. When steam-distilled wood turpentine has been carefully refined so that 90 to 95 per cent of it distills at from 155° to 170° C. (311° to 338° F.), both of these color tests are practically useless.

Turpentine adulterated with more than 10 to 20 per cent of coal-tar oils, or of gasoline or kerosene which have not been deodorized, may usually be readily detected by the characteristic odor of the mixture. The odor of rosin spirits, while quite distinctive, is difficult to detect in mixtures with turpentine. The presence of petroleum oils is also indicated by bubbles or "beads" persisting for a few moments on the surface of the turpentine shaken in a partly filled bottle.

The presence of more than about 10 per cent of kerosene or similar mineral oils is readily detected by the spot which a few drops of the sample placed on white paper leaves on drying. Gasoline and other light mineral oils do not leave this spot.

MINERAL OIL.

It is possible so to adulterate turpentine that neither the specific gravity, refractive index, or flash point is materially altered. Such careful adulteration is, of course, very rare, but as adulterants can not invariably be detected by making these determinations, and further, as mineral oil is the most probable adulterant, it is more rational to at once polymerize the suspected sample, note the volume, color, and consistency of the unpolymerized residue, and determine its refractive index.

The detection of mineral oils in turpentine is relatively quite simple, and is accomplished by mixing the turpentine with a certain proportion of sulphuric acid of a given strength, in which the turpentine is destroyed and mixes with the acid while most of the mineral oil remains unaffected and separates in a layer on top of the acid. The polymerization or sulphonation of turpentine, as this operation is

called, as improved by Mr. Donk and conducted in the Bureau of Chemistry, is as follows:

Place approximately 25 cc of exactly 38 times normal sulphuric acid in a flask holding approximately 50 cc and having a graduated neck. The ordinary Babcock milk-test bottle serves admirably, and being a stock article is readily obtainable. Cool the flask in ice and run in without mixing 5 cc of the turpentine to be tested. If the turpentine is pure and recently distilled, the charred surface will have a green fluorescence when held to the light and the underlying sulphuric acid will be clear and slightly colored. As little as 5 per cent of mineral oil prevents the green fluorescence and gives the sulphuric acid a muddy appearance. Gum and wood turpentine containing considerable quantities of heavy terpenes also lack fluorescence and are muddy in appearance. Mix the turpentine with the acid by cautious shaking, keeping the temperature below 65° C. (149° F.) by frequent immersion in ice. When the temperature no longer increases on shaking (determine by the touch), shake vigorously to insure perfect contact between the acid and the turpentine, place the flask in water at about 40° C. (104° F.), and heat slowly to 65°, shaking vigorously and frequently. When this temperature is attained, give a final vigorous shaking; cool, bring the unpolymerized layer into the neck by running in ordinary sulphuric acid, centrifuge or let stand for two hours, read the volume of unpolymerized clear top layer and immediately determine its refractive index, withdrawing a portion from the top of the clear layer only for this purpose. The character and quantity of this layer or residue will show whether or not the turpentine is adulterated with mineral oil. If the turpentine is pure, the residue will seldom exceed 1 per cent; it will be very viscous, and will have a refractive index at 20° C. of 1.5000 to 1.5200. If the sample contains mineral oil, however, the residue will be limpid or oily and colorless and will have a refractive index at 20° C. between 1.4200 and 1.4950.

It is characteristic of mineral oils that the refractive index of the unpolymerized oil is lower than that of the original sample, while that of the residue from turpentine is higher. The results are not quantitative, as when 5 per cent of mineral oil is present approximately 80 per cent of it is taken up by the sulphuric acid, when 10 per cent of mineral oil is present approximately 60 per cent, and when 20 per cent is present approximately 50 per cent of mineral oil is destroyed in polymerization. When a straight mineral oil is treated with the 38 normal sulphuric acid, about 25 per cent of it is acted upon. For kerosene these figures are much lower. In this report residues greater than 1 per cent are considered to be mineral oil and no correction is made for the amount destroyed in polymerization. These results are therefore low, giving the benefit of the known action of the acid on the mineral oil entirely to the seller.

In a number of instances when the volume of the residue after polymerization did not indicate the presence of mineral oil but its refractive index or limpidity did, the turpentine was distilled, and the portion which did not distill below 170° C., and several of the fractions from the distillation were subjected to the sulphuric-acid test. As a rule, the portion which did not distill below 170° C. left more residue after polymerization than the original turpentine did and it possessed more decidedly the characteristics of mineral oil, i. e., low

refractive index and limpidity. The undistilled portion of pure turpentine when treated in the same way either foamed out of the flask or was instantly reduced to a charred mass and left no measurable residue.

ROSIN SPIRITS.

If rosin spirits is present in notable quantities, it may be detected by the amount of residue on polymerization in conjunction with lowered initial boiling point and the deep coloration produced by mixing portions of the original turpentine or the first fraction with sulphurous acid and with hydrochloric acid.

If the initial distilling temperature of the turpentine is less than 154° C. and the original sample gives a reddish color with sulphurous acid and a greenish red with hydrochloric acid, and the residue from polymerization is less than 5 per cent,¹ it is probable that the sample is adulterated with rosin spirits. An odor of rosin spirits in the sample, or in any fraction, would be confirmatory, and all tests should be repeated on the first fraction, obtained with a fractionating column. At best the detection of small amounts of rosin spirits is very difficult, and the procedure outlined may often fail to detect its presence.

COAL-TAR OILS.

The presence of marked quantities of coal-tar oils, benzol, toluol, and xylol is detected by the odor, the lower initial distilling temperature, i. e., between 80° and 155° C. (176° and 311° F.), and by further examination of the first fractions which distill below 155° C. (311° F.). The rise of temperature on shaking during polymerization is also indicative of the nature of the sample. Coal-tar and petroleum oils do not raise the temperature more than 35° or 40° C. (95° or 104° F.), when mixed with sulphuric acid. When the temperature of turpentine mixed with 38 normal sulphuric acid does not rise above 50° or 60° C. (122° or 140° F.), it is evident that mineral oil, coal-tar oil, or some other oil than turpentine is present in large quantities. The older the sample of turpentine, the greater, or at least the more rapid, the rise of the temperature of the mixture. When the initial distilling temperature of the sample is less than 154° C. (309° F.) and there is less than 1 per cent of residue on polymerization, the presence of coal-tar oils is to be suspected. The sample should be carefully fractionated with a column, the initial distilling temperature noted, and that portion distilling below 157° C. (315° F.) carefully examined. A refractive index at 20° C. greater than 1.475 is strongly indicative of coal-tar oils and the test should be repeated, distilling a sufficient quantity of the sample to obtain at least 50 cc fractionating below 157° C. (314° F.), which should be

¹ Rosin spirits leaves a small residue on polymerisation.

carefully refractionated, the initial distilling temperature noted, and the refractive index, behavior with sulphuric acid (color, heat developed, refractive index of residue, if any), and odor closely observed. An initial distilling temperature of less than 154°C . (309°F .) with a refractive index of the first fraction of more than 1.475, together with a reddish color and but little development of heat on mixing with the sulphuric acid, and the characteristic odor of the oils, is proof of the presence of coal-tar oils.

When the proportion of coal-tar oils is small, this examination may not always give positive evidence of their presence. As little as 5 per cent of xylol, however, may be identified by the following procedure:

To 150 cc of 4 to 1 sulphuric acid, in a separatory funnel, add 100 cc of the suspected turpentine, being careful to prevent a rise in temperature above 40°C . (104°F .). Mix thoroughly, keeping the temperature below 40°C ., draw off the polymerized portion, transfer the unpolymerized portion to a small distilling flask, and distill slowly, changing the receiver when the distillate ceases to be clear, colorless, and limpid. The temperature at this time should not exceed 180°C . (356°F .). A turpentine containing 5 per cent of xylol, when treated in this way, gave about 3 cc distilling below 180°C . (356°F .). The refractive index was 1.475, the odor that of xylol, and on adding nitric acid a strong odor of nitro-xylol was obtained. This treatment of turpentine with dilute sulphuric acid converts it into a heavy oil (with a refractive index of 1.5+), and not more than a few cubic centimeters will distill from the mixture below 300°C . (572°F .). The method has not so far been sufficiently developed to be of quantitative value, so that the proportion of coal-tar oils present can only be approximated within wide limits.

Nonvolatile oils are, of course, readily detected by distillation, and their nature, so far as it may be determined, is learned by the usual methods.

DISCUSSION OF DATA ON UNADULTERATED SAMPLES.

It will be observed in studying the detailed analytical data that a few samples, which undoubtedly do not contain added adulterants, show some inconsistencies among the figures for specific gravity, refractive index, and behavior on distillation. While, as has been mentioned, this fact is partially accounted for by the use of emergent stem thermometers and the variation in barometric pressure, it is certainly not always due entirely to these causes, but also partly to the fact that, in examining so large a number of samples, it was impracticable to make all the determinations on each sample at one time, and changes occurred in these few turpentines in the time that elapsed between the beginning and the ending of their analysis. For some of

the samples two sets of results are given. They show that while some have undergone no alteration even after 18 months others have continued to change. All of the figures on the latter samples are of interest as indicative of the changes which may take place in turpentines during aging. The results on the turpentines which had evidently changed greatly were not considered in forming a judgment regarding the individual constants of turpentine.

The following summary of the analytical data on unadulterated turpentines, based on the detailed figures given in Tables I and II, shows the extremes obtained:

Summary of analytical data on unadulterated turpentines.

Determinations.	Minimum.	Maximum.
Initial distillation temperature ($^{\circ}$ C.).....	154.5	159.0
Distillation below 160° C. (per cent).....	42.0	94.0
Distillation below 165° C. (per cent).....	68.8	98.6
Distillation below 170° C. (per cent).....	77.3	99.1
Specific gravity.....	.8617	.8989
Refractive index.....	1.4684	1.4818
Color (mm).....	2.0	800.0
Polymerization residue (per cent).....	.2	1.0
Refractive index of polymerization residue.....	1.500	1.5150

In setting these limits the results on several samples have been ignored, because there was doubt as to the purity of the sample, or as to the exactness of the results.

The summarized data show that turpentines which are unadulterated, in the sense that they have had nothing added to or taken from them, give widely different results when analyzed under the usual conditions. Thus unadulterated turpentine may begin to distill, under the conditions obtaining in this work, at from 154.5° to 159° C. The amount distilled below 160° C. varies from 42 to 94 per cent, below 165° from 69 to 99 per cent, and below 170° from 77 to 99 per cent. The specific gravity ranges from 0.8617 to 0.8989 at 20° C. and the refractive index from 1.4684 to 1.4818. The residue on polymerization with 38 normal sulphuric acid ranges from 0.2 to 1.0 per cent, and the refractive index of this residue at 20° C. may be as high as 1.5200 but should never be below 1.500. These differences are due to natural variations in the freshly distilled turpentine, to aging, and to variations in conditions of analysis.

It should be borne in mind that many samples of old turpentine are undoubtedly represented in this lot, as well as turpentines stored in wood and in metal, and in clean containers as well as in tanks which still held the residues of previous purchases. In other words, the samples fairly represent those found on the wholesale and retail market at any time. Certain of these samples can not be properly termed turpentine because they do not possess the constants of turpentine.

It has been pointed out that in aging, turpentine undergoes change; it becomes heavier, thicker, and deeper colored and the odor is different. With age specific gravity and refractive index may become higher, the quantity distilling below 170° C. less, the color deeper, and polymerization with sulphuric acid more rapid, and fully as complete. The increase in specific gravity or refractive index which occurs with age is well shown by the results on samples Nos. 6121, 6127, 6135, 6137, and 6238, on which two sets of results, the higher one obtained from 12 to 18 months later than the lower, are given. These samples had visibly changed in consistency and color, and the odor had also changed in the time elapsing between the two sets of determinations. Samples which showed no visible darkening or thickening had practically the same specific gravity and refractive index after 18 months as at first. As has been shown, partly because of this tendency to change with age and also because of the natural variations in the product, it is customary to specify certain limits within which the results of an analysis of turpentine should fall. The fixing of these limits for different grades is discussed under specifications for turpentine.

EXTENT OF ADULTERATION WITH MINERAL OIL.

The data showing the extent and distribution of the adulteration of turpentines, which is practically confined to the addition of mineral oils, may be summarized as follows, the detailed figures being given in Tables I and II, beginning on page 32. Only those samples showing 1 per cent or more of mineral oil are counted as adulterated in this summary:

Number and distribution of adulterated samples.

Source of samples.	Number.	Number adulterated.	Per cent adulterated.	Per cent of adulterant present.
Samples taken in turpentine belt:				
Producers' goods.....	71	3	4.2	2.9
Primary buyers' goods ¹	44	6	13.6	2.2
Dealers' goods.....	99	18	18.0	6.4
Totals and averages.....	214	27	12.6	5.0
Samples taken outside of turpentine belt:				
Primary buyers' goods ¹	7	0		
Dealers' goods.....	205	50	24.4	10.6
Totals and averages.....	212	50	24.0	10.6
General totals and averages.....	426	77	18.0	6.1

¹ Buyers purchasing through factors.

The figures indicate that only a few of the samples of producers' goods are adulterated with mineral oils, while the turpentine of the primary buyers and dealers in the turpentine belt is adulterated extensively, showing 13.6 and 18 per cent of adulterated samples,

respectively. The dealers' samples contain a much higher percentage of the adulterant than either of the other two classes, exceeding the producers' goods by 3.5 and the primary buyers' goods by 4.2 per cent. It will be observed also that adulterations occur more frequently and are present in larger quantities outside the turpentine-producing belt than within it. Eighteen per cent of all the samples collected were adulterated, and the average percentage of adulterant in these was 6.1 per cent, ranging from 1 to 71 per cent in the individual samples. The average amount of mineral oil in a barrel of the adulterated samples was approximately 3 gallons, which, basing the calculation simply on the cost of the two materials, made these adulterated turpentines worth fully \$1.50 less per barrel than unadulterated stock. These facts assume graver significance when it is remembered that turpentine is an article so well known and so generally used that it is carried in stock and sold by merchants generally, particularly by country merchants, and when bought from any source is liable to be employed indiscriminately, either medicinally or technically.

These samples having been carefully taken from all sections of the country, this statement regarding the percentage of adulterated turpentine may be considered as fairly representative of the general conditions in the turpentine trade. The consumption of turpentine in the United States during 1908 was approximately 15,000,000 gallons. If 18 per cent of this was adulterated, then approximately 3,000,000 gallons of adulterated turpentine, containing an average of at least 6 gallons per hundred of mineral oil, costing about one-fifth as much as the turpentine, was sold at turpentine prices. The small percentage of mineral oil found in some samples may have been occasioned by mixing a barrel of heavily adulterated turpentine with a large quantity of a pure product, or possibly by putting turpentine into improperly prepared petroleum barrels or into tank cars previously used for mineral oils.

EXAMINATION OF STANDARD GRADE SAMPLES.

The proper grading of turpentine is a matter of no small monetary importance to the producer and primary buyer, and the boards of trade of the turpentine markets have adopted rules and standards for the purpose of correct and consistent grading. These standards are selected by the naval stores inspectors and passed upon by the naval stores committee of the boards of trade. The generally accepted final authority is the New York naval stores committee, whose inspector, as a matter of fact, selects practically all standard samples of turpentine and rosin used by naval stores inspectors of this country.

For the purpose of learning how closely the standard samples on which turpentine is bought in the primary markets agree among themselves, sets of standards were secured from the Savannah, Ga., and

Jacksonville, Fla., boards of trade and also from the inspectors of the New York naval stores committee.

The color of these samples was read against the standard yellow glass on the Lovibond tintometer, marked "1." The turpentine was placed in a graduated 200-mm tube and the depth of the column of turpentine required to equal the color of the glass was carefully noted, using the Schreiner type of colorimeter. The longer the column of turpentine required to match the glass, the less color there is in the turpentine. The results are expressed in millimeters, which show the length of column or depth of turpentine required to equal the standard yellow glass No. 1. The following results were obtained on these standard samples:

Comparison of standard turpentine samples from different sources.

Sample marked—	Savannah sample.	Jacksonville samples.		New York sample.
		No. 1.	No. 2.	
	mm.	mm.	mm.	mm.
Water white.....		192	180	150
Standard.....	¹ 180	40		50
Off 1 shade.....	36	26	33	25
Off 2 shades.....	16	22	20	12
Off 3 shades.....		4	12	6
Off 4 shades.....	4			4

¹ This sample probably should have been marked "water white."

These results show that the standard samples obtained from different sources are far from uniform and indicate different practices on the chief primary markets. It is the usual custom, however, as previously stated, to mix small quantities of off-shade turpentine with the standard or water-white products and thus the off-shade output is used and sold without getting beyond the primary markets. The variations noted in the standards have, therefore, but little significance in the retail trade where, in general, but one grade of turpentine is recognized, though, as will be seen by consulting the tables, there is a great variation in the color of commercial turpentine, which point as an indication of quality should undoubtedly receive more careful consideration from the consumer than it does.

PROPOSED SPECIFICATIONS FOR TURPENTINE.

Buyers of large quantities of turpentine have long specified certain tests with which the turpentine they purchased must comply. In the main, most of these are reasonable and will be met by carefully made turpentine when freshly distilled. The constants and standards thus set differ slightly in different specifications, and some of them are unnecessarily severe, causing the rejection of much good turpentine. It seems well, therefore, to suggest specifications for

turpentine, which, while fully protecting the consumer, recognize the natural variations occurring even in the freshly distilled turpentine.

This investigation, however, shows clearly that by no means all of the unadulterated turpentine sold can be regarded of the same quality, nor does it comply with the accepted standards. This fact is fully accounted for by natural differences in the crude turpentine, negligence on the part of the turpentine producer, irregularities in distilling, and differences in age and methods of storing. A turpentine with a specific gravity of 0.875, only 50 per cent of which distills below 160° C. and 20 mm of which equals in color the No. 1 Lovibond glass, is certainly not of the same grade as one having a specific gravity of 0.865, 90 per cent distilling below 160° C. and 150 mm of which is needed to equal the No. 1 glass in color. Nevertheless the former is turpentine, though its value is somewhat less than the latter. It appears but just and reasonable that several well-defined grades of turpentine should be recognized and standard specifications established for them. Such standards are accordingly suggested for the consideration and criticism of producers and consumers of turpentine.

Standard or No. 1 turpentine should have a specific gravity at 20° C. of from 0.862 to 0.870; a refractive index at 20° C. of from 1.468 to 1.476; 95 per cent should distill below 170° C., and a layer of not less than 200 mm should be required to equal in color the Lovibond yellow glass No. 1. On polymerization with 38 normal sulphuric acid the residue should not exceed 1 per cent, should be reddish in color and viscous, and its refractive index at 20° C. should be from 1.500 to 1.520. An unadulterated turpentine which does not agree with these requirements may properly be regarded as not of standard or No. 1 quality.

Second quality or No. 2 turpentine should have a specific gravity at 20° C. of from 0.862 to 0.875; a refractive index at 20° C. of from 1.468 to 1.480; 90 per cent should distill below 170° C. and a depth of not less than 100 mm should be required to equal the Lovibond yellow glass No. 1. The polymerization residue must not exceed 1 per cent and must have a refractive index of not less than 1.50.

Third quality or No. 3 turpentine should have a specific gravity at 20° C. of from 0.865 to 0.880; a refractive index at 20° C. of from 1.468 to 1.485; 60 per cent should distill below 170° C. and a depth of not less than 50 mm should be required to equal the Lovibond yellow glass No. 1. The polymerization residue must not exceed 1 per cent and must have a refractive index of not less than 1.500.

These specifications are applicable to both gum spirits turpentine and to "wood turpentine," and are in general harmony with trade practice. For high-grade varnishes and for medicinal purposes a colorless, light turpentine which evaporates rapidly and will hasten drying, leaving the varnish firm and not "tacky," is desired. For

lower-grade varnishes and good quality paints a somewhat colored and slower evaporating product may be successfully employed. In the case of low-grade paints, especially those where the drying is hastened by vigorous means, or in all cases when slow drying is desired, the heavier, slower evaporating turpentine, or even what is known as "pine oil," may prove most suitable.

METHODS FOR THE EXAMINATION OF TURPENTINE FOR PURITY AND QUALITY.

The work done in connection with this investigation has developed a number of improvements in the methods for examining turpentine which increase the accuracy of the results and insure greater concordance in the work of different chemists. The methods to be given have been in use in this laboratory for from one to three years and have been found satisfactory.

All tests of a turpentine should be promptly made, usually within the same week, because some samples change rapidly in consistency, color, gravity, behavior on distillation, and refractive index, and if the several determinations are made at long intervals those last made may not represent the turpentine as first received. Further, all of the results on a sample may not be strictly comparable and may lead to errors in judging the turpentine.

COLOR.

Determine color in a graduated 200 mm tube in a colorimeter reading against the Lovibond yellow glass No. 1. If a depth of 200 mm or more is required to match the glass the color is No. 1; if a depth of from 100 to 200 mm is required the color is No. 2; if a depth of from 50 to 100 mm is required it is No. 3; and if less than 50 mm the color is No. 4.

SPECIFIC GRAVITY.

Determine specific gravity with a pycnometer, plummet, Westphal balance, or accurate hydrometer. The determination may be made at 15.5° or at 20° C., as desired.

DISTILLATION.

APPARATUS.

Heating bath.—Use a copper cup of about 800 cc capacity, having a concave tight-fitting cover (which may be fixed) with a hole in it large enough to hold a 300 cc distilling flask, and also a one-half inch condensing tube 12 inches long, rising from one side of the cup. This serves as a condenser for the vapors of glycerin or oil used in the bath, and prevents the superheating effect of vapors which would otherwise be in contact with the exposed portion of the distilling flask.

Distilling flask.—Comparable results can only be obtained in distillation by always using flasks of the same dimensions. The flask found most satisfactory in this work is an ordinary 300 cc flask, 8 cm in diameter, with the side tube 8 cm from the main bulb, and the neck extending 8 cm above the side tube. The neck is 2 cm in diameter and the side tube is 5 mm.

Manometer.—An open manometer with a movable scale graduated in millimeters, and provided with double connections, must also be provided.

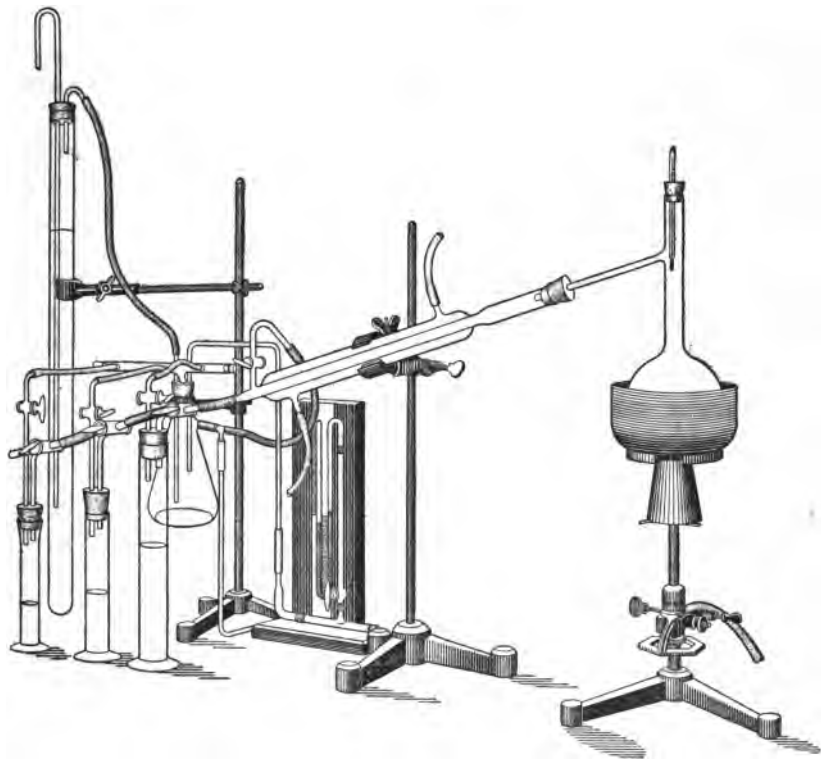


FIG. 1.—Apparatus for the fractional distillation of turpentine.

Pressure regulator.—This consists of a glass tube about 1 inch in diameter and about 24 inches long, fused at one end, nearly filled with water, and stoppered with a two-hole stopper, carrying two glass tubes, one of which extends to the bottom of the large tube, the other being free to move up or down.

Condenser and receivers.—An ordinary condenser is connected with three stoppered, graduated cylinders, by means of two-way stopcocks, care being taken to insure close connection and the exposure of a minimum length of tubing to the turpentine. The stopper of each cylinder is provided with a stopcock connection with an air chamber,

which is in turn connected with the pressure regulator and with the manometer. By connecting a blast or vacuum with the air chamber, which in turn is connected with the free end of the proper tube of the pressure regulator any desired pressure (within the limits of the apparatus) can be produced in the system. The apparatus is shown in figure 1. All corks and connections must be absolutely air tight. If they are not, the fact will be indicated by the sudden fluctuation of the thermometer when the cock between a receiving cylinder and the pressure flask is opened.

OPERATION.

Place 200 cc¹ of the turpentine and several small pieces of pumice in the distilling flask, connect with the condenser, set the distilling flask in the bath, and heat. Stopper the distilling flask with a cork through which passes a standardized thermometer graduated from 150° to 200° C. (302° to 392° F.). The mercury bulb is placed opposite the side tube and the 175° C. (347° F.) mark must be below the cork. Open the stopcock from the air chamber to the first cylinder and also the two-way cock connecting the condensers with the first cylinder, in order that the condensed turpentine may flow into it. According as the pressure is above or below 760 mm connect the pressure regulator with the vacuum or the blast and manipulate the regulator until the manometer indicates in millimeters the reduced or added pressure needed to bring the pressure within the flask to 760 mm.² Heat the bath slowly, and when distillation begins regulate the heat so that the turpentine distills at the rate of 2 drops per second. When the thermometer reaches 160° C. (320° F.) close the stopcocks to the first cylinder and open those to the second. When the thermometer reaches 165° C. (329° F.) close the stopcocks to the second cylinder and open those to the third. When the temperature reaches 170° C. (338° F.) discontinue distillation. Measure or weigh each fraction and determine its specific gravity and refractive index. A polymerization test is also made on each fraction and the residue when it appears advisable.

When the residue not volatile at 170° C. is large, and it is desired to continue distillation for any purpose, a clean cylinder may be put in the place of the first or second one and distillation continued as long as is deemed necessary. Of course as many fractions may be made as are desired, one with each change of 1° C. in the distillation temperature, or with the distillation of definite volumes. Such a procedure, however, adds but little, if any, information of technical value.

¹ The sample and the fractions may be weighed if preferred.

² In exact work the barometer should be corrected to 0°.

REFRACTIVE INDEX.

Determine the refractive index with a Zeiss direct reading refractometer at 20° C.

DETECTION OF ROSIN SPIRITS.¹

Note the initial distilling temperature and to one portion of the first fraction distilling below 160° C. (320° F.) add sulphurous acid and to another hydrochloric acid. An initial distilling temperature below 154° C. (309° F.), together with the development of a decided red color with sulphurous acid and a green color with hydrochloric acid, when accompanied by a polymerization residue of less than 5 per cent on the original sample, is indicative of rosin spirits. Refractionate 50 to 100 cc of the first fraction, distilling below 160° C. (320° F.) with a column, note the initial distilling temperature, and repeat the color and polymerization tests. If they are more positive than before, rosin spirits is probably present.

DETECTION OF COAL-TAR OILS.¹

To 150 cc of sulphuric acid (4:1) in a separatory funnel, add 100 cc of the suspected turpentine, being careful to prevent a rise in temperature above 40° C. Mix thoroughly, keeping the temperature below 40° C. (104° F.), draw off the polymerized portion, transfer the unpolymerized portion to a small distilling flask, and distill slowly, stopping the distillation when the temperature reaches 180° C. (346° F.), or before if the distillate becomes cloudy, colored, or oily. Determine the refractive index, which should be 1.475 or more, and add cautiously a little nitric acid. The odor of nitrobenzol or nitro-xytol proves the presence of coal-tar oils.

DETECTION AND ESTIMATION OF MINERAL OIL.

The method used for the detection and estimation of mineral oil is an improvement of the Hertzfeldt method of polymerization with sulphuric acid and is based on the fact, observed by Mr. Donk, that the residue from turpentine is practically constant and negligible if the acid used is of a certain exact concentration. The method has been in use in this laboratory for about three years with perfectly satisfactory results.

Preparation of acid.—The sulphuric acid used is 38 times normal. This solution contains 100.92 per cent of sulphuric acid by weight and is prepared by adding fuming acid to the concentrated until a strongly fuming mixture is obtained. The exact strength is then determined by diluting and titrating a weighed portion (6 to 8 grams), using methyl orange as indicator. An accurate determination of the acid strength is absolutely necessary, as an acid contain-

¹ See page 19 for further details as to the determination of these adulterants.

ing 100.82 per cent or 37.9 normal gives a residue decidedly greater than one containing 100.92 per cent of sulphuric acid, when the mineral oil present is 5 per cent or less. The acid is standardized as follows:

The acid is conveniently weighed in a bulb having a capillary tube at the lower end and a rather large tube with a glass stopcock in the upper end, fitted with a platinum wire for weighing. The pipette is filled by aid of a slight vacuum, and by closing the stopcock simultaneously with the withdrawal of the capillary from the acid the lower end of the capillary is emptied and may be cleaned on the exterior by wiping first with a wet and then with a dry piece of cloth. The pipette thus filled and dried may be left for hours without losing or gaining weight.

After weighing, dip the capillary into about 500 cc of water in a large beaker, open the stopcock, and when the acid has run out, wash the pipette with water run in from the top through a rubber tube and funnel. Add a slight excess of sodium carbonate solution of known strength by weight (0.2 gram per gram of solution is convenient); this may be run in from a suitably arranged dropping or delivery flask (weighed before and after the operation) until the solution is slightly alkaline to methyl orange. Boil off the carbon dioxid, allow to cool, add methyl orange, and titrate back the excess carbonate with half normal sulphuric acid. Determine the specific gravity of the acid with a pycnometer, calculate its normality, and run in from a burette, having a long capillary delivery tube, enough water to dilute it to 38 normal.

Other procedures may suggest themselves and be preferred by different operators, but of the many methods of standardization tried, this one was found to be by far the most accurate and as expeditious as any, ordinary care holding the error below one-tenth of 1 per cent. It is needless to say that the acid must be carefully guarded against absorbing moisture from the air. Several liters of the acid should be prepared at a time and it should be stored in quarter or half liter bottles closed with tight-fitting glass stoppers.

Operation.—Transfer 20 cc of this acid to a Babcock bottle (one having a 20 cc mark is convenient), stopper, and place in ice water. After cooling, add 5 cc of the turpentine, again cool, and gradually mix the contents, cooling from time to time (the temperature should not be allowed to rise materially), and when the mixture no longer warms up after shaking, agitate thoroughly. Then place the bottle in a water bath in which the water is on a level with the acid and heat to from 60° to 65° C. (140° to 149° F.) in the course of about five minutes, keeping the contents thoroughly mixed by vigorously shaking six or seven times. Do not stopper the bottles after turpentine has been added.

(As the destruction of the turpentine depends upon its contact with the sulphuric acid, which in turn is dependent upon the minuteness of the turpentine particles suspended in the acid, it follows, and this is convincingly borne out by experience, that thorough mixing is one of the salient features of the method.)

Cool to room temperature, add ordinary sulphuric acid until the contents rise in the graduated portion of neck, and whirl at 1,200 revolutions per minute for three or four minutes, or allow to stand over night; read the amount of supernatant residue.

Discussion.—With a pure turpentine the residue will be straw colored, viscous, and if read at once will give a refractive index at 20° C. of 1.5050 or higher and amount to from 0.02 to 0.05 cc. When adulterated with mineral oil the residue is a limpid, colorless liquid, having a refractive index of 1.4900 to 1.4300 or lower, depending on the kind and amount of adulterant present. When a small amount of adulterant has been added it is preferable to use a centrifugal machine reading the refractive index immediately, using the upper portion of the residue, as the mineral oil and residual turpentine are thus stratified, the low-reading mineral oil constituting the upper portion. A capillary pipette is used in transferring a small portion of the residual oil to the refractometer.

The results are always low, as members of the unsaturated aliphatic series are partly soluble in the acid used. When a mineral oil of an asphaltum base has been used as an adulterant, results are usually approximately too low by from 80 to 50 per cent. That is the percentage of mineral oil present that is polymerized by acid of the strength employed or that remains mixed with the acid is higher with low percentages of mineral oil and lower with high percentages. The method has been in use in this laboratory for several years in connection with pure food and drug work as well as in testing turpentine, paint, varnish, oils, etc., bought under Government contracts, and an experienced operator following the method strictly has no difficulty in detecting 2 per cent of adulterant.

TABULATED DATA ON COMMERCIAL SAMPLES.

TABLE I.—Samples collected in the turpentine-producing States.

(L=limpid; O=oil; V=viscous; WW=water white.)

PRODUCERS' SAMPLES.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color. mm.	Initial distilling temper- ature. ° C.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Polymerization.		Minimum per cent of mineral oil present.
									Per cent residue.	Refrac- tive index of residue.	
1543	Albany, Ga.	Wood turpentine.	0.8680	1.4694	220	157	97.0	99.0	1.04(V)	1.5044	None.
3449	New Orleans, La.		.8822	1.4719	400	157	95.0	97.5	1.4	1.5100	Do.
6066	do.			1.4721	60	157	96.4	98.0	1.8	1.5023	Do.
6084	do.		.8905	1.4747		156.4			.4(V)	1.5098	Do.
6090	do.		.8698	1.4708	280	155.4	94.0		.6		Do.
6091	do.		.8807	1.4722		155	96.0		.4(V)		Do.
6094	Mobile, Ala.		.8686	1.4718	300	157	94.0				Do.
6096	do.		.8796	1.4725							Do.
6098	do.		.8782	1.4720	22	156	94.5		.4(V)		Do.
6099	do.		.8658	1.4706	300	150.4	96.0		.8		Do.
6100	do.		.8785	1.4717	450	156	95.0		.4(V)		Do.
6101	do.		.8696	1.4713	280	155	94.0				Do.
6115	Pensacola, Fla.		.8841	1.4715	360	156	96.0				Do.
6117	do.		.8700	1.4701	110	156.4	95.0	98.6	.6(V)		Do.
6120	do.		.8665	1.4691	300	156	98.1	96.5	.4(V)		Do.
6124	do.		.8666	1.4691	120	155.4	94.2				Do.
6127	Jacksonville, Fla.		.8745	1.4705	280	156.4	95.0		.4(V)		Do.
6128	do.		.8679	1.4683	360	157	95.2	97.8			Do.
6132	do.		.8702	1.4702	280	156.4	96.1	97.9	.2		Do.
6133	do.		.8661	1.4692	212	156	95.9	98.1	.4		Do.
6134	do.		.8712	1.4713	450	156.4	96.2	98.4	.2		Do.
6135	do.		.8740	1.4706	320	157	95.1	97.6	.6		Do.
6137	do.		.8862	1.4728	10	156	96.7		.4	1.5060	Do.
6138	do.		.8757	1.4709							Do.
6139	do.		.8627	1.4683	30	157	98.7	92.4	.2(V)		Do.
6140	Savannah, Ga.		.8657	1.4703	240	156	96.9				Do.
6141	do.		.8657	1.4692	140	156.4	97.9				Do.
6142	do.		.8736	1.4715	113	156	94.0	97.3			Do.
6143	do.		.8742	1.4718	WW	156.4	97.7	98.5			Do.

6156	do.	1.4710	8646	225	156	98.0	93.7	.4	Do.
6157	do.	1.4699	8671	250	156	90.1	97.9	.2(V)	Do.
6158	do.	1.4705	8705	250	156	95.9	96.7	.4(V)	Do.
6159	do.	1.4719	8779	300	157	90.7		.2	Do.
6160	do.	1.4698	8667	600	156	98.6			Do.
6200	do.	1.4707	8724	230	156	98.3			Do.
6201	do.	1.4710	8702	350	157	95.0	97.9	.4	Do.
6223	Jacksonville, Fla.	1.4712	8743	94	157	97.7			Do.
6224	do.	1.4712	8703	157	156	87.2	95.4	.4(V)	Do.
6225	do.	1.4697	8631	600	156	97.6		.2(V)	Do.
6226	do.	1.4697	8657	450	156	93.0			Do.
6227	do.	1.4705	8696	500	157	96.0			Do.
6228	do.	1.4712	8635	500	157	93.9			Do.
6229	do.	1.4686	8730	460	156	95.8	98.2	.4	Do.
6230	do.	1.4700	8672	204	157	96.4	93.3	2.2(L)	2.2
6231	do.	1.4694	8665				98.1	.4	None.
6232	do.	1.4721	8736	157	157	97.2	98.3	.4	Do.
6233	do.	1.4691	8689	132	157	93.7	97.6	.4	Do.
6234	do.	1.4706	8671	300	157	93.9			Do.
6235	do.	1.4702	8793	200	156	98.3	93.9	2.0(L)	1.4598
6235	do.	1.4715	8702	360	156	92.0	95.0	.4(V)	None.
6236	Brunswick, Ga.	1.4704	8698	350	157	96.4	98.3	.4(V)	Do.
6237	do.	1.4705	8770	600	156	97.7		.2	Do.
6238	do.	1.4732	8699	300	157	94.5	97.9	.2	Do.
6242	do.	1.4712	8708	300	157	94.4	97.7		Do.
6243	do.	1.4686	8660	550	156	98.3	98.9		Do.
6244	do.	1.4699	8685	400	157	95.0	98.1		Do.
6245	do.	1.4704	8680	346	156	96.3	98.0		Do.
6311	Fernandina, Fla.	1.4715	8708	350	157	94.0	97.7		Do.
6404	do.	1.4711	8676	500	158	95.0	98.4	.6(V)	Do.
6407	do.	1.4697	8666	238	157	95.8	98.2	.4(V)	Do.
6408	do.	1.4705	8673	258	157	97.4	98.7	.2	Do.
6409	do.	1.4713	8755	440	156	97.5		.6	Do.
6410	New Orleans, La.	1.4715	8763	240	157	95.2	98.6	1.4(L)	Do.
6554	do.	1.4728	8747	156	156	59.7	80.6	1.4950	Do.
6555	do.	1.4728	8736	500	156			.6(V)	Do.
6556	Original barrel.	1.4717	8703	340	156	94.7	97.2	.4(V)	Do.
6557	do.	1.4713	8725	360	156	98.7		.6(V)	Do.
6558	do.	1.4702	8677	400	157	97.2	98.2	.2(V)	Do.
6559	do.	1.4733	8771	33	158	56.7	97.8	.8(O)	Do.
6570	Wood turpentine.	1.4706	8692	300	156	96.2	97.8	.2(V)	Do.
6571	Pure spirits turpentine.	1.4705	8692	300	156	96.2	97.8	.2(V)	Do.
6574	do.	1.4694	8638	460	156	77.1	84.3	1.2	Do.
6576	Covington, La.	1.4694	8671	270	156	70.2	86.1	4.4(L)	Do.
6578	New Orleans, La.	1.4693	8671	270	156	70.2	86.1	4.4(L)	Do.
6596	do.	1.4703	8687	400	157	96.2	97.6	.4(V)	4.4

* Redetermined 18 months later.

* Determinations made 12 months later.

* Distillation residue on polymerization gave 0.0 per cent.

* Below 160° C.

* Rejected; 3 shades off.

TABLE I.—Samples collected in the turpentine-producing States.—Continued.
PRIMARY BUYERS' SAMPLES.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distilling temper- ature. °C.	Per cent distilling residue 165° C.	Per cent distilling residue 170° C.	Polymerization.		Minimum per cent of mineral oil present
									Per cent residue.	Refrac- tive index of residue.	
6065	New Orleans, La.	Industrial purposes.	0.8319	1.4676	mn. 107	1554	82	90	5.6	1.4528	5 &
6068	do.					156	904	85.5	5.8 (O)	1.4793	1.8
6069	Gulfport, Miss.			1.4747	20	156	94	Top tank	1.8	1.4745	1.8
6068	do.			1.4761	20	1554	93	Bot. tank	1.8	1.4773	1.2
6069	do.					156	91	86	1.6 (L)	1.4780	1.2
6069	New Orleans, La.			1.4712	418	1554	91				1.2
6069	do.			1.4760	320	1554	91				1.2
6069	Mobile, Ala.			1.4768	320	1554	90				1.2
6069	do.			1.4708	260	1554	83				1.2
6067	do.			1.4708	260	1554	83				1.2
6116	Pensacola, Fla.			1.4695	400	156	93.8	97.6	4 (V)		Do.
6121	do.			1.4742		156	96.1	97.3	.6	1.5080	Do.
6122	do.			1.4738	82	156	95.3	97.3			Do.
6123	do.			1.4721	300	156	95.3	98.9	4 (V)		Do.
6125	Mobile, Ala.			1.4721	300	156	96.0		4 (V)		Do.
6129	Jacksonville, Fla.			1.4686	30	156	95.0		4 (O)	1.5010	Do.
6130	do.			1.4768	28	156	91.2	93.9	4 (V)	1.5070	Do.
6148	Savannah, Ga.			1.4710	130	1564	94.4	96.3	.2		Do.
6149	do.			1.4713	200	157	93.8	96.9	2 (V)	1.5100	Do.
6150	do.			1.4717	350	157	92.2	96.6			Do.
6151	do.			1.4758	85	156	93.5	96.9	.2		Do.
6239	Brunswick, Ga.			1.4758	85	156	92.0	95.9			Do.
6240	do.			1.4712	180	1574	92.3	97.7	2 (V)		Do.
6241	do.			1.4710	200	1574	92.3	97.7	2 (V)		Do.
6241	do.			1.4712	200	1574	95.5	98.3	4 (V)		Do.
6246	do.			1.4725	240	157	98.9				Do.
6247	do.			1.4704	280	157	93.2	98.0	2 (V)		Do.
6247	do.			1.4700	736	156	94.8	97.3			Do.
6248	do.			1.4774	14	156	71.8	85.5			Do.
6249	do.			1.4733	2	157	86.3	91.9	2 (V)	1.5000	Do.
6405	Fernandina, Fla.			1.4701	260	157	94.3	97.3			Do.
6405	do.			1.4696	180	157	94.9	97.6	4 (V)		Do.
6414	do.			1.4725	360	157	95.3	97.7	4 (V)		Do.
6415	do.			1.4725	42	1564	88.7	95.1	2 (V)		Do.
6552	do.			1.4723	WW				2 (O)	1.5094	Do.
6553	do.			1.4696	WW				2 (O)	1.4704	Do.
6558	Wilmington, N. C.			1.4717	104				2 (O)	1.4791	Do.
6575	New Orleans, La.			1.4710	158	157	91.5	95.5	.4 (O)	1.4980	Doubtful trace.

TABLE I.—Samples collected in the turpentine-producing States—Continued.

DEALERS' SAMPLES.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive Index.	Color. mm.	Initial distilling temper- ature. ° C.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Polymerization.		Minimum per cent of mineral oil present.
									Per cent residue.	Refrac- tive Index of residue.	
3420	Richmond, Va.	Spirits of turpentine.	0.8638	1.4709	80						None.
3421	Fayetteville, N. C.	Turpentine.	.8676	1.4665	110						Do.
3422	do.	Spirits of turpentine.	.8667	1.4702	66						Do.
3423	Hartsville, S. C.	Turpentine.	.8628	1.4686	80						Do.
3424	Florence, S. C.	do.	.8750	1.4715	100						Do.
3425	Hartsville, S. C.	do.	.8659	1.4754	4						Do.
3426	Florence, S. C.	Spirits of turpentine.	.8623	1.4686	100						Do.
3429	Georgetown, S. C.	do.	.8753	1.4663	34						Do.
3430	Fayetteville, N. C.	do.	.8788	1.4720	24						Do.
3432	Rocky Mount, N. C.	Turpentine.	.8696	1.4707	84						None.
3433	Florence, S. C.	Spirits of turpentine.	.8740	1.4729	14						Do.
3434	do.	Turpentine.	.8650	1.4691	69						Do.
3435	do.	Spirits of turpentine.	.8639	1.4691	600						Do.
3436	Georgetown, S. C.	do.	.8617	1.4692	260						None.
3437	Fayetteville, N. C.	do.	.8679	1.4703	112						Do.
3438	Rocky Mount, N. C.	Turpentine.	.8646	1.4703	70						Do.
3439	do.	do.	.8697	1.4709							Do.
3440	do.	do.			36						Do.
3442	do.	do.	.8724	1.4690							Do.
3444	do.	do.	.8723	1.4710	100						Do.
3445	Florence, S. C.	Virgin spirits.									Do.
3446	Lithington, N. C.	Turpentine.	.8714	1.4693	31						Do.
3448	Rocky Mount, N. C.	do.	.8718	1.4685	32						Do.
6069	New Orleans, La.	do.	.8706	1.4697	66						Do.
6070	Gulfport, Miss.	do.	.8669	1.4707	180						Do.
6071	do.	do.	.8662	1.4715	330						Do.
6080	New Orleans, La.	do.	.8614	1.4678							Do.
6082	do.	do.	.8621	1.4732	152						Do.
6083	do.	do.	.8790	1.4726	100						Do.
6085	Gulfport, Miss.	do.	.8724	1.4710	300						Do.
6086	do.	do.	.8720	1.4652	2						Do.
6113	New Orleans, La.	do.	.8722	1.4700	126						Do.
6114	Pensacola, Fla.	do.	.8689	1.4701	360						Do.
6118	do.	do.	.8640	1.4688	330						Do.
6119	do.	do.			156						Do.
6126	Jacksonville, Fla.	do.	.8697	1.4704	30						None.

6131	do.	1.4704	.8662	190	157	94.8	98.6	2	Do.
6136	do.	1.4740	72	156	84.5	80.1	2.8(L)	Do.
6138	do.	1.4710	.8677	27	153	77.1	88.5	2.8(L)	2.8.
6139	do.	1.4752	.8897	44	157	86.7	94.1	2(V)	None.
6140	do.	1.4711	.8740	120	160	52.7	74.5	4(V)	Do.
6141	do.	1.4712	.8848	80	158	66.0	80.1	.4	Do.
6142	Tallahassee, Fla.	1.4786	54	157	68.8	77.3	2(V)	Do.
6143	do.	320	157	97.9	97.9	2(V)	Do.
6144	do.	1.4762	6	155	78.1	86.8	2	Do.
6145	do.	1.4755	100	157	94.9	97.3	2	Do.
6146	do.	1.4725	.8747	310	157	94.1	97.8	4(V)	Do.
6147	do.	1.4710	.8690	200	157	96.7	98.1	Do.
6308	Brunswick, Ga.	1.4740	.8784	84	157	88.0	94.3	4(V)	Do.
6309	do.	1.4720	.8715	44	157	82.5	89.1	2.4(V)	Do.
6310	do.	1.4712	.8763	360	157	88.4	93.1	4(V)	Do.
6318	do.	1.4713	.8757	200	156	94.2	96.7	2(V)	Do.
6368	Technical purposes, guaranty No. 1083, strictly pure spirits of turpentine.	1.4690	.8698	70	156	95.7	98.5	2(L)	Do.
6369	do.	Do.
6370	do.	1.4733	.8678	300	152-157	93.9	96.7	Do.
6371	do.	1.4790	.8707	54	156	96.1	98.5	2(V)	Do.
6411	Fernandina, Fla.	1.4709	.8652	260	156	92.0	96.3	4(V)	Do.
6412	do.	1.4698	.8681	4(V)	Do.
6413	do.	1.4717	.8701	159	4(V)	Do.
6559	Greenville, Miss.	1.4708	.8676	200	157	88.1	94.9	2.6(L)	Do.
6560	do.	1.4703	.8628	130	157	92.3	96.1	4(O)	None.
6562	do.	1.4747	.8664	44	156	94.9	96.4	4(V)	Do.
6563	do.	1.4708	.8640	360	156	97.7	Do.
6567	New Orleans, La.	1.4659	.8543	163	157	57.0	73.9	12.4	Do.
6568	do.	1.4795	150	157	92.0	94.9	4.4(O)	Doubtful trace.
6569	do.	1.4672	.8570	170	158	71.7	81.8	12.0(L)	12.0.
6571	do.	1.4667	.8576	300	156	63.2	78.9	9.6(L)	9.6.
6572	do.	1.4684	.8600	200	135-4	78.7	88.7	8.0(L)	8.0.
6573	do.	1.4720	.8698	60	157	94.1	96.9
6577	do.	1.4713	.8767	180	157	83.6	90.9	4.8(L)	4.8.
6580	do.	1.4691	.8641	110	157	80.1	90.1	3.6(L)	3.6.
6581	do.	1.4726	.8825	600	156	96.9	98.5	4(V)	None.
6582	do.	1.4732	.8636	77	160	30.9	66.3	1.0(O)	Do.

1 Distillation residue on polymerization gave 0.4 per cent (L); refractive index, 1.4502.

2 Distillation residue on polymerization gave 0.0 per cent.

3 Distillation residue on polymerization gave 3.6 per cent (L); refractive index, 1.4470.

4 Distillation residue on polymerization gave 61.2 per cent (L); refractive index, 1.4340.

5 Wood turpentine, steam distilled.

6 Distillation residue on polymerization gave 0.8 per cent (L); refractive index, 1.4570±.

TABLE I.—*Samples collected in the turpentine-producing States—Continued.*

DEALERS' SAMPLES—Continued.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distilling temper- ature.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Polymerization.		Minimum per cent of mineral oil present.
									Per cent residue.	Refrac- tive index of residue.	
6583	New Orleans, La.8880	1.4738	mm. 160	157	88.9	94.5	1.4(O)	1.4900	Doubtful trace.
7005	North Fort Worth, Tex.8097	1.4703	150	156 $\frac{1}{2}$	91.3	95.0	.4	1.4955	None.
7006	Fort Worth, Tex.8672	1.4698	260	155 $\frac{1}{2}$	93.1	95.4	3.4(O)	1.4946	Doubtful trace.
7009	do.	Pure spirits of turpen- tine.	.8908	1.4736	40	157	85.3	91.3	.6(V)	None.
7010	Birmingham, Ala.8897	1.4734	300	156	92.7	96.1	.6	Do.
7011	do.8770	1.4698	90	155	83.3	96.9	.6	Do.
7013	Waco, Tex.8650	1.4701	270	Do.
7217	Austin, Tex.8646	1.4729	110	156	95	99 $\frac{1}{2}$	2(V)	1.5024	Do.
7315	San Antonio, Tex.	(¹)	.8680	1.4705	260	155	92	95	2.2(O)	1.5050	Do.
7317	do.8724	1.4710	140	154 $\frac{1}{2}$	92	95	4.6(O)	1.4986	0.6.
7318	do.8608	1.4751	170	150 $\frac{1}{2}$	55	75	1.1(O)	1.4977	Doubtful trace.
9152	Houston, Tex.	Pure wood turpentine; not for medicinal use.	60	157	92.7	97.7	.8	1.5055	None.
9175	do.	Turpentine.	.8643	1.4688	714	1.5075	Do.
9340	New Orleans, La.	Pure spirits of turpen- tine.	.8659	1.4699	804	1.5129	Do.
9641	El Paso, Tex.	Oil turpentine; spirits of turpentine.	.8694	1.4701	1254	1.5090	Do.
9642	do.	(Bottle) Climax pure turpentine; not for medicinal use.	1006	1.5027	Do.
9722	Gainesville, Tex.	Pure turpentine; not for medicinal use.	.8750	1.4704	80	1.2	1.4820	1.2.
9723	Denison, Tex.	Pure spirits of turpen- tine.	.8700	1.4700	1008	1.5072	None.
9726	Sherman, Tex.	Turpentine.	.8672	1.4705	954	1.5080	Do.
9761	do.	do.	.8661	1.4700	1304	1.5080	Do.
9931	Fort Worth, Tex.	do.	.8663	1.4700	1208	1.5088	Do.
9981	do.	Pure spirits of turpen- tine.	.8652	1.4695	704	1.5066	Do.
10504	El Paso, Tex.	Climax pure turpen- tine; not for mediet- nal use.	.8669	1.4699	1504	1.5060	Do.

No.	Locality.	Quantity.	Weight.	Specific gravity.	Do.
10865	Victoria, Tex.	Pure spirits of turpen- tine.	.8685	1.4700	.4
11086	Brenham, Tex.	Strictly pure turpen- tine.	.8646	1.4704	.4
12961	West Point, Miss.	Turpentine.....	.8714	1.4710	1.2
12982	do.	Spirits of turpentine...	.8680	1.4708	.4

Distillation residue on polymerization gave 0.4 per cent (L); refractive index, 1.4739.
 Distillation residue on polymerization gave 0.8 per cent (L); refractive index, 1.4520.
 Distillation residue on polymerization gave 0.0 per cent (L).
 Wood turpentine.
 Distillation residue on polymerization gave 0.02 per cent (V O); refractive index, 1.4630.
 Distillation residue on polymerization gave 0.12 per cent (L); refractive index, 1.4472.
 Distillation residue on polymerization gave 0.02 per cent (L); refractive index, 1.4840.

TABLE II.—Samples collected outside the turpentine-producing States.

PRIMARY BUYERS' SAMPLES.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distilling tempera- ture.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Polymerization.		Minimum per cent of mineral oil present.
									Per cent residue.	Refrac- tive index of residue.	
3447	St. Paul, Minn.	Refined turpentine....	0.8988	1.4688	mm. 360	°C. 155	91.0	95.5	1.8(O)	1.4994	None, Do. but full trace.
5376		Industrial purposes only.									N.D.
6382	Minneapolis, Minn.	Do.	.8763	1.4716	300	156	92.9	98.4	.6		N.D.
6384	Chicago, Ill.	Turpentine....	.8910	1.4738	190	156½	96.5	98.5			Do.
6401	Clinton, Iowa.	Industrial purposes only.	.8994	1.4708	228	156	92.2	96.5			Do.
6419	Oklahoma City, Okla.	Pure spirits of turpen- tine.	.8655	1.4697	450	155½	95.5	96.5	.4		Do.
6546	Kansas City, Mo.	Technical use....	.8706	1.4706	360	155	95.9	97.8	.4(V)		Do.

¹ Distillation residue on polymerization gave 0.4 per cent (O); refractive index, 1.4556.

TABLE II.—*Samples collected outside the turpentine-producing States—Continued.*
DEALERS' SAMPLES.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color. mm.	Initial distilling temper- ature. ° C.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Polymerization.		Minimum per cent of mineral oil present.
									Per cent residue.	Refrac- tive index of residue.	
2989	Washington, D. C.	Turpentine.	0.8761	1.4712	90	<1	None.
2990	do.	do.	0.8742	1.4723	60	Do.
2991	do.	do.	1.4852	12	3	1.4850	2.0.
2992	do.	do.	0.8688	1.4708	112	<1	None.
2993	do.	Spirits of turpentine.	0.8890	1.4703	80	<1	Do.
2994	do.	Turpentine.	0.8863	1.4734	54	<1	Do.
2995	do.	do.	1.4818	18	<1	Do.
2996	do.	do.	0.876	1.4879	6	1.4(L)	1.4651	1.4.
2997	do.	do.	<1	None.
2998	do.	do.	0.8730	1.4712	144	<1	Do.
2999	do.	do.	0.8892	1.4747	100	<1	Do.
2100	do.	do.	0.8790	1.4725	200	<1	Do.
2101	do.	do.	0.8679	1.4707	130	<1	Do.
2102	do.	Pine spirits	0.8176	1.4472	34	77.6(L)	1.4405	77.6.
3419	Hartsville, S. C.	Turpentine.	0.8634	1.4693	60	<1	None.
3426	Petersburg, Va.	do.	0.8659	1.4701	34	<1	Do.
3427	do.	do.	0.8664	1.4707	100	<1	Do.
3431	do.	Spirits of turpentine.	2.0(L)	1.4829	2.0.
3440	Richmond, Va.	Turpentine.	0.8630	1.4684	50	<1	None.
3441	Petersburg, Va.	do.	0.8656	1.4695	54	<1	Do.
3443	Richmond, Va.	Spirits of turpentine.	0.8659	1.4698	44	<1	Do.
6290	Washington, D. C.	Wood turpentine.	0.8658	1.4708	96	48.7	73.1	4(V)	1.50+	Do.
6291	do.	Webb's excelsior tur- pentine.	0.8623	1.4724	52	90.2	96.2	4	Do.
6292	do.	Pure spirits of turpen- tine.	0.8871	1.4710	300	98.2	98.9	4	Do.
6293	do.	Turpentine.	0.8897	1.4724	200	<1	Do.
6294	do.	Spirits of turpentine.	0.8765	1.4707	450	91.6	98.8	<1	Do.
6306	St. Joseph, Mich.	do.	0.8674	1.4694	360	97.9	98.7	4(Y)	Do.
6307	do.	do.	0.8681	1.4702	120	95.8	97.3	Do.
6373	Minneapolis, Minn.	Destructive.	152-160	92.5	94.1	1.4(L)	1.4983	Do.
6374	St. Paul, Minn.	Pure gum spirits.	0.8747	1.4715	200	84.5	90.9	1.0(O)	1.4430	Doubtful, trace.
6375	do.	Rectified turpentine.	0.8688	1.4700	280	96.7	98.0	1.4(Y)	1.4324	1.0.
6376	Minneapolis, Minn.	Industrial purposes only.	0.8708	1.4710	180	96.9	98.7	12.0(L)	1.4324	None.
6379	St. Paul, Minn.	Rectified oil of turpen- tine.	1.4690	88	96.7	97.7	1.4(V)	1.5079	None

TABLE II.—*Samples collected outside the turpentine-producing States—Continued.*
DEALERS' SAMPLES—Continued.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distilling temper- ature. ° C.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Polymerization.		Minimum per cent of mineral oil present.
									Per cent residue.	Refrac- tive index of residue.	
6551	Philadelphia, Pa.	Pure spirits of turpen- tine, guaranteed.	0.8672	1.4704	80	157	96.9	97.9	28.0(L) .4	1.4512	28.0.
6561	Baltimore, Md.	Bought as pure.	.8650	1.4714	56	157	95.9	98.1	.2	None.
6587	Titusville, Pa.	Pure spirits of turpen- tine.	.8684	1.4720	157	157	94.9	97.4	.4(V)	Do.
6588	Franklin, Pa.	Pure spirits of turpen- tine.	.8672	1.4692	170	157	91.2	95.7	1.2(L)	1.4620	Do.
6589	San Francisco, Cal.	Pure spirits of turpen- tine.	.8786	1.4733	242	156	93.2	96.7	1.4(L)	1.4876	1.2.
6591	do.	Not for medicinal use.	.8585	1.4679	85	158	72.9	83.4	7.6(L)	1.4435	Trace.
6592	do.	Spirits of turpentine.	.8716	1.4713	180	155	93.9	96.6	.2	7.6.
6593	do.	Technical use.	.8704	1.4717	50	157	89.7	94.2	.2(V)	None.
6594	do.	Pure spirits of turpen- tine.	.8704	1.4717	50	157	89.7	94.2	Do.
6595	Omaha, Nebr.	Pure spirits of turpen- tine.	.8661	1.4705	400	159	92.0	96.5	1.0(O)	1.4630	1.0.
6596	do.	Bought as pure.	.8712	1.4718	103	159	70.5	89.2	1.4(L)	1.4508	Trace.
6597	do.	Industrial purposes.	.8739	1.4702	79	156	78.7	88.3	4.8(O)	1.4532	4.8.
6598	do.	Billed and shipped as turpentine.	.8670	1.4703	232	156	94.3	96.9	.4(L)	1.4785	Trace.
6599	do.	Industrial purposes.	.8693	1.4709	200	157	92.8	96.0	.2(V)	Do.
6600	do.	do.	.8689	1.4702	334	158	45.1	67.1	.4(V)	Do.
6601	Beellingham, Wash.	Spirits of turpentine.	.8698	1.4710	140	156	91.9	95.1	.4(V)	Do.
6602	do.	Not for medicinal use.	.8579	1.4738	28	159	89.7	94.0	.4(O)	1.5010	Do.
6603	San Francisco, Cal.	Pure spirits of turpen- tine.	.8698	1.4685	190	157	73.5	88.9	6.8(L)	1.4410	6.8.
6604	do.	Turpentine.	.8705	1.4721	180	155	89.9	93.4	.4(V)	1.5060	None.
6605	do.	Turpentine.	.8677	1.4692	156	156	92.3	96.0	Do.
6606	do.	Not for medicinal use.	.8706	1.4720	140	157	89.9	94.2	.2(V)	1.5060	Do.
6607	do.	Pure spirits of turpen- tine.	.8734	1.4705	98	156	91.1	95.3	.2(V)	1.5064	Do.
6608	do.	For internal use.	.8673	1.4740	4	156	91.8	95.5	Do.
6609	do.	Pure eastern spirits of turpentine.	.8777	1.4720	200	157	71.1	80.5	9.6(L)	1.4375	9.6.
6610	do.	For internal use.	.8663	1.4684	90	157	93.5	96.1	.4(L)	1.4925	<1.
6611	do.	Pure eastern spirits of turpentine.	.8755	1.4710	180	156	93.5	96.1	<1.
6612	Buffalo, N. Y.	Pure spirits of turpen- tine.	.8755	1.4710	180	156	93.5	96.1	<1.

TABLE II.—*Samples collected outside the turpentine-producing States—Continued.*

DEALERS' SAMPLES—Continued.

L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color.	Initial distilling temper- ature ° C.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Polymerization.		Minimum per cent of mineral oil present.
									Per cent residue.	Refrac- tive index of residue.	
8001	Little Rock, Ark.	Technical purposes only.	0.8655	1.4686	mm.	155½	96.2	97.7	.4(V)	1.5106	None.
8002	do.	Spirits of turpentine, guaranty No. 453.	.8651	1.4688	70	156	96.6	98.0	.4(V)	1.5090	Do.
8013	Lawrence, Mass.	Spirits of turpentine.		1.4459					Trace.		27.
8015	Las Vegas, N. Mex.	do.							1.5	1.4699	1.5.
8685	Terre Haute, Ind.	Strictly pure turpen- tine.							22.4	1.4483	22.4.
8819	New Bedford, Mass.	Spirits of turpentine.							3.6	1.4680	3.6.
8946	Shelbyville, Ind.	Spirits of turpentine.							1.6	1.5077	None.
9036	Prescott, Ariz.	Pure spirits of turpen- tine.							1.0	1.5074	Do.
9037	do.	Pure spirits of turpen- tine.							16.4	1.4135	16.4.
9038	do.	Pure spirits of turpen- tine.							.6	1.5069	None.
9039	Seattle, Wash.	Pure spirits of turpen- tine.							.8	1.4860	Doubtful
9050	Huntington, W. Va.	Pure spirits of turpen- tine for technical use.							3.2	1.4584	3.2.
9051	Williamson, W. Va.	Pure spirits of turpen- tine.							.4	1.5075	None.
9150	Bluefield, W. Va.	Spirits of turpentine.			37		87.4	92.6	.8	1.5082	Do.
9151	Decatur, Ind.	Pure spirits of turpen- tine.			70		91.5	95.1	.4	1.5085	Do.
9153	Bluefield, W. Va.	Pure spirits of turpen- tine.			70	157			2.8	1.4645	2.8.
9177	Salem, Va.	Turpentine for indus- trial use.			88				.4	1.5130	None.
9201	Covington, Ky.	Pure turpentine.			100				2.8	1.4555	2.8.
9202	Detroit, Mich.	Strictly pure turpen- tine.			85	155	87.0	94.0	.4	1.5112	None.
9203	Cincinnati, Ohio.	Pure spirits of turpen- tine for industrial purposes.			70				.4	1.5090	Do.
9252	Houston, Tex.	Pure spirits of turpen- tine.			120				5.0	1.4551	5.0.
9306	Tucson, Ariz.	Strictly pure turpen- tine.	.8606	1.4672	90				.4	1.5100	None.
9339	Oklahoma City, Okla.	Spirits of turpentine.	.8658	1.4700	135						

9450	Tucson, Ariz.....	Pure spirits of turpen- tine.....	1.4630	80	5.2	1.4520	5.2
9459	Charleston, W. Va.....	do.....	.8636	170	1.6	1.4748	1.6
9513	Washington, D. C.....	do.....	1.4692	110	1.5125	None.
9523	Sumner, Pa.....	Spirits of turpentine.....	.8662	140	6	1.5095	1.4
9558	Sharonkin, Pa.....	Pure spirits of turpen- tine.....	1.4692	65	1.4	1.4742	1.4
9595	Tucson, Ariz.....	do.....	1.4669	54	7.2	1.4450	7.2
9644	Buffalo, N. Y.....	do.....	1.4710	105	4	1.5100	None.
9691	Portland, Ore.....	Turpentine.....	1.4705	120	1.5046	Do.
9697	Pueblo, Colo.....	do.....	1.4705	116	8	1.5052	Do.
9883	Oklahoma City, Okla.....	Spirits of turpentine.....	.8668	140	4	1.5100	Do.
10008	Cincinnati, Ohio.....	Strictly pure turpen- tine.....	1.4712	38	4	1.5110	Do.
10009	Tampa, Fla.....	Turpentine.....	.8656	60	4	1.5115	Do.
10010	do.....	Pure spirits of turpen- tine.....	1.4690	220	4	1.5100	Do.
10095	Tucson, Ariz.....	Pure spirits of turpen- tine for technical purposes.....	1.4684	200	71.0	1.4430	71.0
10201	Strictly pure turpen- tine.....	1.4512	60	4	1.5095	None.
10257	Newcastle, Pa.....	Pure turpentine.....	.8630	50	165	45	1.4670	Do.
10469	New York, N. Y.....	do.....	1.4690	200	2.0	1.4850	2.0
10505	Gallatin, Tenn.....	Pure spirits of turpen- tine.....	1.4695	80	19.5	1.4311	19.5
10575	do.....	.8661	4	1.5110	None.
10633	Waterbury, Conn.....	Turpentine.....	1.4700	Do.	Do.
10647	do.....	Pure turpentine.....	1.4650	166	84	1.4510	9.4
10648	do.....	do.....	1.4702	138	96	1.5116	None.
10649	do.....	Pure wood spirits.....	1.4672	101	45	68	1.4576	3.8
10650	do.....	Wood spirits.....	1.4735	173	5.6	1.4539	5.6
10651	do.....	Chemically pure wood spirits.....	.8662	170	4	1.5069	None.
11000	Clarksville, Tenn.....	Strictly pure turpen- tine.....	1.4704	180	8	1.5116	Do.
11001	Bowling Green, Ky.....	Pure spirits of turpen- tine.....	1.4688	80	5.7	1.4560	5.7
11065	Boulder, Colo.....	Superior quality tur- pentine.....	1.4708	250	4	1.5110	None.
11101	Turpentine.....	WW	158	90	3.0
11102	do.....	do.....	WW	159	None.
10103	do.....	do.....	.8661	WW	158	2.0	2.0
11168	Pure turpentine ¹	do.....	1.4710	162	18	71	1.5055	None.
11216	do.....	Pure turpentine.....	1.4708	159	86	92	Do.
11380	do.....	do.....	1.4700	Do.	Do.
11381	Washington, D. C.....	do.....	.8678	159	86	93	Do.	Do.
12468	Cincinnati, Ohio.....	Turpentine.....	1.4709	159	88	93	1.5112	Do.
12511	do.....	do.....	1.4705	1.0	Do.
12540	do.....	do.....	.8690	Do.	Do.
12705	Washington, D. C.....	do.....	1.0	1.5135	Do.

Destructively distilled wood turpentine:

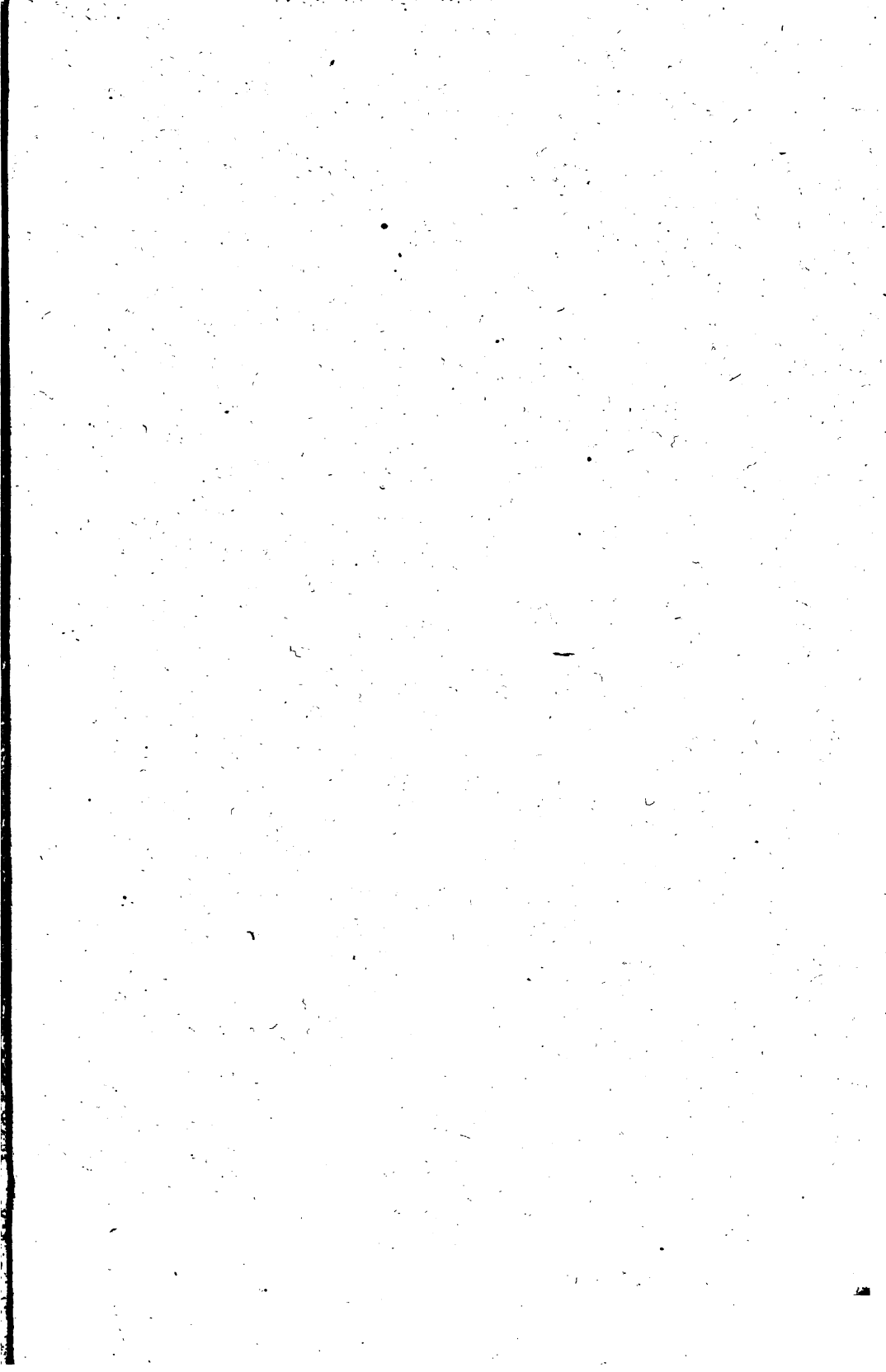
TABLE II.—*Samples collected outside the turpentine-producing States—Continued.*

DEALERS' SAMPLES—Continued.

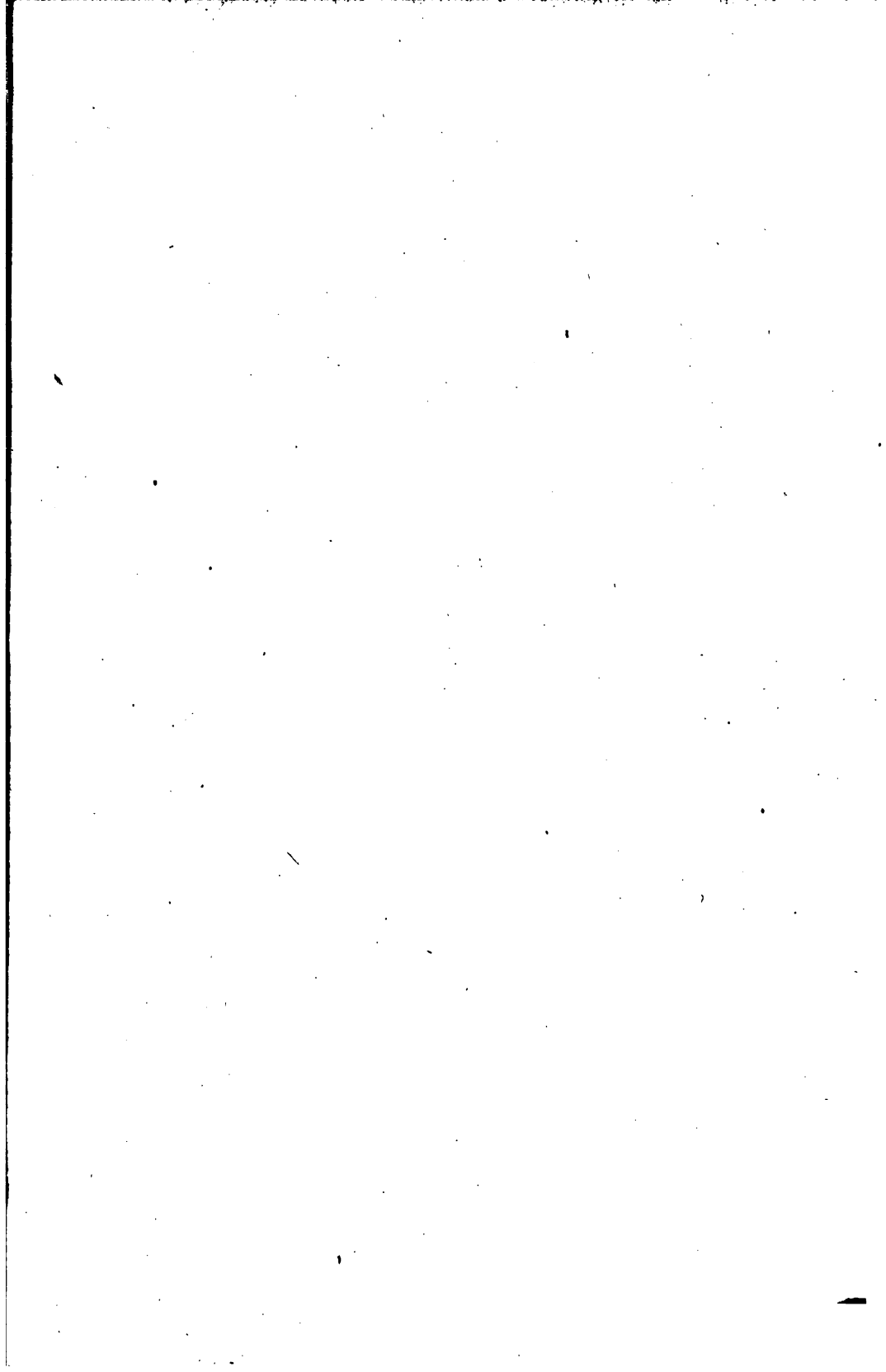
L. and P. No.	Sampled at—	Sample labeled or sold as—	Specific gravity.	Refrac- tive index.	Color. <i>mm.</i>	Initial distilling temper- ature. ° C.	Per cent distilling below 165° C.	Per cent distilling below 170° C.	Polymerization.		Minimum per cent of mineral oil present.
									Per cent residue.	Refrac- tive index of residue.	
12710	Clarksville, Tenn.	Pure turpentine.....	.8660	1.4705					19.4	1.4500	19.2
12712	Washington, D. C.	Turpentine.....	.8535	1.4645					18.6	1.4505	18.6
12713	do.	do.							1.2	1.5050	None.
12875	Washington, D. C.	do.							1.0	1.5090	Do.
12940	do.	Pure turpentine.....	.8639	1.4700					.8	1.5150	Do.
13033	do.	do.	.8650	1.4708					.8	1.5115	Do.
13130	Danville, Ky.	Spirits of turpentine....	.8777	1.4713	160				.4	1.5140	Do.
13131	Cincinnati, Ohio	Pure brand turpentine....	.8673	1.4712	80				.8	1.5150	Do.
13167	Washington, D. C.	Pure turpentine.....							.4	1.5150	Do.
13168	do.	do.	.8641			159	90.7	96.7	.5	1.5146	Do.
13205	Springfield, Mass.	Pure spirits of turpen- tine.	.8613	1.4652	80				8.4	1.4715	8.4
13529	Douglas, Ariz.	Pure turpentine.....	.8646	1.4698	65				1.2	1.5100	None.
13672	Waterbury, Conn.	Pure spirits of turpen- tine.	.8596	1.4690	120				5.6	1.4600	5.6
13673	Washington, D. C.	Turpentine.....	.8637	1.4700	200				1.0	1.5120	None.

O













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